

City of Houston
INTER OFFICE CORRESPONDENCE

From: Controlled Substances Section ~ROM: James T. Miller, Criminalist Specialist
Crime Laboratory Crime Laboratory
To: J.K. Alexander, Criminalist Lab Manager Date: February 11, 2010
Crime Laboratory
C.1 ~ zn ~
Re: Lori Wilson, Criminalist Lab Manager Subject: Clarification of Reporting Guidelines
Criminalist Laboratory
Imma Rios, Assistant Director
Crime Laboratory

In the 02-01-10 Controlled Substances S4P revision there was an error in the Reporting Guidelines section. Effective immediately this section will read:

p.3 Reporting Marijuana, Marijuana Seeds and Hash
4. For cases that consist of marijuana seeds
this may be reported as "Marijuana seed:
and the weight in ounces. If no seed, report as "No controlled substance
identified" with a footnote: "Marijuana were identified and determined to be
incapable of beginning germination."
In addition, the Reporting Guidelines
6 to state the following:

If a substance has been sufficient to preliminary pharmaceutical identification
without analytical confirmation the report should reflect "Indication [substance]"
with an asterisk.

Indication Amitriptyline - Dangerous drug *
Indication Acetaminophen - Over the counter *

* Pharmaceutical identification only
It was not the intent of this modification to require the notation "Pharmaceutical
identification only"
but to provide it as an optional addition on the report. This memo will serve to revise the
section to
read as follows:

If a substance has been subjected to preliminary pharmaceutical identification
without analytical confirmation, the report will reflect "Indication [substance]".
If a dangerous drug or over the counter substance is indicated, then the report will
include the notation that the substance is a dangerous drug or an over the counter
product. The notation "Pharmaceutical identification only" may be added as in

~

the following example:

Example: Indication Amitriptyline - Dangerous drug *
Indication Acetaminophen -- Over the counter *

*

Pharmaceutical identification only

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James T. Miller, Criminalist Specialist~

John T. Miller, Criminalist Laboratory

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CITY OF HOUSTON

INTER OFFICE CORRESPONDENCE

To: Controlled Substances Section
Crime Laboratory
K. Alexander, Criminalist Lab Manager
Crime Laboratory

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~ y ~ Lori Wilson, Criminalist Lab Manager
~ Crime Laboratory . ~ ~ ~ ~ ~ Z Or ~,; ~ ~ < ~.

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Ircna Rios, Assistant Director
Crime Laboratory
Effective 04-01-10 the following footnote will be added to all cases:

The Houston Police Department Crime Lab
total weight of the substances and has retained
under the provisions of chapter 481.164 of
excess quantities will be destroyed
Laboratory receives notice from the
Houston Police Crime Laboratory ' r'r
disposition of the narcotic(s).

Sections CS-SOP 16 Excess Quantity
to reflect this modification.

jtm:jtm .a
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nd CS-SOP 19 Reporting Guidelines have been re~ised

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James T. Miller, Crimnirialist Specialist
Crime Laboratory

FROM: 3ames T. Miller, Criminalist SpecialistCrime Laboratoryoa,rE: March 22, 201~

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separate. notifzcation unless the~rney's off ce before that date. Thecient documentation as to
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CITY 4F HOUSTON

INTER OFFICE CORRESPONDENCE

From: Controlled Substances Section From: James T. Miller, Criminalist Specialist

Crime Laboratory Crime Laboratory

To: K.K. Alexander, Criminalist Lab Manager Date: March 19, 2010

, Crime Laboratory

Re: v

Re: Lori Wilson, Criminalist Lab Manager Subject: Quality Checks for Reagents and Spot Plates

Crime Laboratory ~ 3~y~ ~ I p

Irma Rias, Assistant Director

Crime Laboratory

In order to improve the Controlled

Substances Section the following sections:

CS-SOP 03

CS-SOP 05

CS-SOP 12

CS-SOP 13

CS-SOP 14

Attached are copies of April 1, 2010.

list Specialist

jtm:jtm

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H~USTON POLICE DEPARTMENT
CRIME LABORATORY

DOCl~iEIVTi ~-U~HORIZATION

Requestor: James T. Milfer Date:_ Jan 20. 2010

~X~ Existing Version: CS-SOP Version 2009 revised for 2010 effecti~e 02-01-10

C~ New If new document, brief~y describe document contents.

Submit hardcopy or electronic documentation to the Quality Ass ance Manager

Signature: / ~%/~ A ate. ~-t~--a _

~ ~ Approve 1 ~ ~ Re~ect Date:~.~_

~ ~ Appro~e / ~ ~ Reject Date:

~ ~ A~pro~e / ` ~ Reject Date:

Comments:
QA Mana4e~ Review
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~ f~ Approve 1 ~ ~ . "" L.. ~ ~ ~ ;'~; ~ "" _Date: ~ ~ 7 ` ~~~`~ ~ ~ --

Pian for implementation:

Comments:

Laborato Director A rovai
~ ~ Approve / ~ ~ Reject Date:
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STANDARD OPERATING PROCEDURES

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. ~ SUPPORT OP~RATIONS

~'~'~•~'~ CRIME LABORATORY DEVISION

CONTRQLLED SUBSTANCES SECTION

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• Rescinded 06-01-04

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STANDARD OPERATING PROCEDURES

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~~ SUPPORT OPERATIONS

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4pT'~ ~ CRIM~ LAB~RATORY ~IVISION

CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE ISSUED: EFFECTIVE DATE: PROCEDURE NUMBER

01-~1-04 02-41-10 CS-SOP 0~

SECTION: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

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SUBJECT/EVENT:

GOALS AND OBJECTIVES

The primary goal of the Crime Laboratory Controlled Substances Section is to support the mission of the Houston Police Department by providing analysis of evidence received for the presence of controlled substances, drugs, and other chemical substances as efficiently as possible utilizing available resources.

To maximize efficiency, cases submitted will be reviewed and the case status identified as Active (on-going investigations, cases pending), Disposed (court accepted plea, submitted for destruction), or Dismissed charges, charges dismissed by the court). Evidence associated with active cases is the primary focus of the section and will be handled based upon the following objectives:

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All priority items should be analyzed as soon as they are received and completed before the end of the year.

. Botanical samples should be dried and analyzed within one week.

. All active essential controlled substance cases should be analyzed within two weeks.

. Fifty percent of all active controlled substance cases should be analyzed and completed within two weeks.

. Seventy-five percent of all active controlled substance cases should be analyzed and completed within thirty days.

. All active controlled substance cases should be analyzed and completed within sixty days.

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All reports should be entered into OLO as soon as possible after the completion of a case but within two working days. This objective is contingent upon the Incident Report having already been entered into OLO.

REFERENCES:

HPD GO 100-06 ; CRIM~ LABORATORY SOP, OBJECTIVES SECTION 14N

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Al) case files should be technically reviewed within two working days following the completion of the report.

All evidence should be returned to Centralized Evidence Receiving (CER) within three working days of the completed case file review.

- The time between receipt of evidence by an analyst and the return of that evidence to CER should be less than one month.

REFERENCES:

HPD GO 100-06 ; CRIME LABORATORY SOP, OBJECTIVES SECTION

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~ GOALS AND OBJECTIVES CS-SOP 01 Page 3 of 3

MODIFICATION SUMMARY

DATE VERSION CHANGE

01-01-09 2009 New format# for Headers and Footers

Add References

p. ~— Third bullet point# change "bulky" to "excess quantity"

p. 1- Seventh bullet point add "This objective is

continued on... in to OL~."

~2-01-10 2010 No changes

`1~

REFERENCES:

HPD GO 100-Q6 ; CRIM~ LABORATORY SOP. OBJECTIVES SECTION

STANDARD OPERATING PROCEDURES

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CRIME LABORATORY DIVISION

CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE ISSUED: EFFECTIVE DATE: PROCEDURE NUMBER

0~01-04 02-01-10 CS-SOP 02

S~CT10N: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

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SUBJECT/EVENT:

EVIDENCE HANDLING

SCOPE ~

Controlled Substances Section.

To provide guidelines for the handling of evidence in

SUBMISSION OF EVIDENCE

following places:

Narcotic evidence may be submitted for analysis

. Through the offsite lock boxes at locations

. In person at the laboratory centralized Evidence Receiving

EVIDENCE RECEIVING (CER)

EVIDENCE HANDLING BY

CER personnel receive substance evidence into the laboratory, enter case related information into the Evidence Management System (EMS), and store the evidence until it is transferred to a analyst. When the analyst has completed work on a case, the evidence is returned to CER personnel to be handled according to CER SOP.

RECEIVING EVIDENCE

It is the responsibility of the analyst to maintain the integrity of the evidence at all times while in his/her custody. All evidence must be protected from loss, cross-transfer, contamination and/or deleterious change.

All evidence received by a drug analyst is to be assigned by the Section Manager or designee and must be documented as follows:

(1) Each proximal container (bag, envelope, box, etc.) must be marked with a unique case identifier (either the assigned incident number or laboratory number) and the analyst's initials. The proximal container is usually a Houston Police Department Evidence Envelope, but it can be anything that contains exhibits for a case. In

REFERENCES:

HPD GO 700-1

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In addition, an item designator may be used with the incident or laboratory number to distinguish items within a case.

(2) A submission form must be filled out for all evidence submitted. If the officer has not attached a submission form to the evidence, a submission form is filled out with all pertinent information available.

(3) All exhibits should be inventoried and compared with the documentation on the submission form. The analyst will itemize the actual evidence received on the Controlled Substances Examination Sheet. If there are significant discrepancies between the submission form and the exhibits themselves, such as missing exhibits, notify your supervisor immediately who will then notify the HPD Narcotics Captain. Generally, these cases will be investigated in-house with notification going to the submitting officer and the Lieutenant if necessary. A supplement should be entered into ORO by the analyst documenting the facts surrounding the case at the conclusion of investigation.

(4)

The information on the submission form should match the information written on the evidence envelope. If the suspect names do not match, the officer will be contacted. He/she may have inadvertently used forms with other evidence, and the mistake needs to be corrected before the case is analyzed. Always notify your supervisor about major discrepancies.

(5)

All exhibits contained within could be labeled with the analyst's initials and the unique case identifier designators. In a case with numerous small items analyzed to such as small ziplocks, the exhibits may be placed in a container such as one in which the analyst has placed the unique case identifier and the name of the analyst and his/her initials. If during the screening tests a difference is noted, the small items will be grouped appropriately and analyzed and labeled sequentially.

CASES CONTAINING CURRENCY, VALUABLES, LARGE ITEMS, AND BULLETS

(1)

All U.S. currency, valuables, large items, and bullets should be transferred to the Property Room, unless they are returned to the submitting officer. Do not write on currency to allow its eventual return to general circulation. Record the serial number(s) or photocopy any paper U.S. currency. In a case with numerous bills, recording the serial numbers may be suspended.

(2) Supplement Property Room transfers in the OLO computer system.

CASES REQUIRING EXAMINATION FOR LATENT PRINTS

Police officers or Assistant District Attorneys (ADA's) may request that any or all items in a case be examined for latent prints. If a case has already been analyzed for controlled

REFERENCES:

HPD GO 7Q0-1

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When the print request is made, the analyst informs the person making the request that the evidence has already been handled. If they still want the case printed, the

Latent Print Lab will be contacted with a request for examination.

If a request for latent prints is made on the submission form, the Latent Print Lab is contacted before any evidence is handled or analyzed. If handling is necessary before a latent print examination, ALWAYS wear gloves and handle the evidence as little as possible. All evidence is inventoried at the time of receipt even when latent print examination is requested.

CASES CONTAINING POSSIBLE BIOHAZARDS

Cases that contain items that could represent a possible biohazard require special handling. While working with possible biohazards, cautions should be taken including wearing gloves, lab coat, and safety glasses taking extra care not to touch any part of your body, especially your face. Work area should become contaminated, wash the area thoroughly with bleach. Avoid touching uncontaminated surfaces (such as telephones, etc.) with soiled gloves. If you work in the hood, clean thoroughly with dilute bleach when you are finished. Whenever possible use disposable beakers, pipette tips, etc. and dispose in the biohazard container. Anything that is not disposable come in contact with bodily fluids needs to be washed with a solution of dilute bleach is prepared by mixing one part commercial bottled bleach to nine parts water. Some items that require are the following:

(1)

Syringes - remove needles with the needle cutters.

(2)

Latex pellets ingested or removed from the stomach or lower bowel - in the hood wash the pellets with a bleach solution while wearing double gloves. After preliminary weighing and sampling of the pellet contents is done in the hood. When you are finished and cleaning the pellets, place them in a ziplock bag. Clean the hood area with dilute bleach solution.

{3}

Items contaminated with blood or items identified as removed from a body cavity, the toilet, groin, crotch area, etc. could represent a biohazard and should be handled accordingly.

SUBMITTING EVIDENCE TO CER

(1)

Repackage all evidence in the same condition it was received whenever possible. If a flammable substance is found in a matchbox inside a ziplock, repackage it as you found it. Do not change the condition of the evidence unless it is absolutely necessary. For example, liquid in an open soda can will be transferred to a jar that can be sealed.

REFERENCES: '
HPD GO 700-1

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(2)

Before sealing evidence for submission to CER, double check that all evidence is properly labeled.

(3) Seal evidence (including analyst initials and date) according to CER sectional SOP

and submit to CER.

DOCUMENTATION OF EVIDENCE TRANSFER

Any evidence that is transferred to a division or agency outside the Crime Laboratory should be documented in OLO. Some examples of situations that should be documented are:

(1)

Jewelry, money, suitcases, bullets, etc. sent to the Property Room.

(2)

Any evidence turned over to another agency (UC, EA, HCOC task force, etc.) for storage.

(3) Any evidence released to an officer for return to the Property Room.

The documentation may be entered into the same supplement as the results of the analysis, or at a later date with the beginning of a new supplement.

An exception to the requirement is the release of controlled substance evidence for court and not Print Lab for further testing.

REFERENCES:

HPD GO 700-1

SUBJECT: EVIDENCE HANDLING CS-SOP 02 Page 5 of 5

MODIFICATION SUMMARY

DATE: 02-01-09

VERSION: 2Q09

CHANGE: New format for Headers and Footers

p. 2- Receiving Evidence (2) delete "See the... chain of custody."

p. 2- Cases Containing Currency... (2) delete "Follow the guidelines... Training Guide."

p. 3- Cases Requiring Examination for Latent Prints delete "Refer to the Training Guide for detailed information."

p. 3- Cases Requiring Examination for Latent Prints delete "To fill out the form, refer to the Training Guide."

p. 3- Cases Requiring Examination for Latent Prints combine last two paragraphs

Add Reference

02-01-10

2020 Change "lab number" to "unique case identifier" throughout 0-09 memo)

p. 1— Evidence Handling by CER

Receiving Evidence (1) delete "If the evidence is

different from other... after the previous receiver's initials."

Cases Containing Currency (1) delete "Do not write "###" on currency..."

p. 3-- Cases Requiring Examination for Latent Prints

First paragraph modify "...the Latent

Print Lab will be contacted with a request for examination.

Delete last paragraph " , ~ ...

p. 4— OLO Documentation of Evidence Transfer delete

"(4) Evidence released for an identification officer for further testing." and add to the last sentence "An exception to the requirement for OLO documentation is the release of controlled substance evidence to court and to the Latent Print Lab for further testing."

REFERENCES:

WPD GO 700-1

STANDARD OPERATING PROCEDURES

1. SUPPORT OPERATIONS
2. CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE ISSUED: EFFECTIVE DATE: PROCEDURE NUMBER

01-41-04 04-01-10 CS-SOP 03

SECTION: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:
04-Q1-10 9 Page 1 of 91
SUBJECT/EVENT:
ANALYSIS GUIDELINES
SCOPE

To provide guidelines for the analysis of controlled substances and dangerous drugs.
PROCEDURE

Note: Only one case shall be opened for analysis. If the case cannot be completed, it must be secured before a case may be opened (e.g. If you have a priority case that requires immediate). This is to ensure that all cases are protected from loss, cross-contamination.

The general guidelines for when an active case needs analysis are as follows (see the Objectives Section for definition of active cases):

- If the charge is possession of a Controlled Substance (PCS) or Delivery of a Controlled Substance (DCS), analyze the highest per se felony substance for each suspect list for felonies, misdemeanor substances and/or residues may be retained and analyzed.

If the charge is DCS/PCS and there is powder or chunk substance labeled as delivery and a residue labeled as possession, then the residue should also be analyzed.

If the charge is PCS and only residues are present then at least one residue per suspect should be analyzed.

If the charge is PCS and only misdemeanor substances are present, then analyze the controlled substances present but retain any dangerous drugs (definition of dangerous drug = prescription drugs not listed in any Schedule or Penalty Group).

If the charge is "obtain drugs by fraud, possession of a dangerous drug, delivery of

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a dangerous drug, practicing dentistry/medicine without a license, fraudulent prescription, etc.," then at least one dangerous drug should be analyzed.

- If the charge is "possession of a dangerous drug" and there are both controlled substances and dangerous drugs present, analyze the controlled substances and retain any dangerous drugs without analysis.

•

Any items in a case indicated as being seized due to a delivery transaction should be analyzed.

•

If there are multiple suspects listed on the submission form, it may be necessary to analyze more items than those outlined above. Check a source of information.

•

In each case, the most significant items should be identified and analyzed.

Considerations must be given to the information provided on the Evidence

Submission Form, or available through OL4. This includes such things

as the specific charges or types of offense due to a single suspect, the examinations requested, the description of evidence submitted, as well as the analyst's visual inspection of the items.

•

Items which are not analyzed may be entered and reported as "Retained with no analysis".

•

If an analyst consults with the District Attorney, or an attorney with the Grand Jury and the evidence items are needed for prosecution, then all other items in a case are retained without analysis. Document the conversation and maintain the connection with the case file.

•

In all cases require analysis of unanalyzed items by a principal associated with a case may require further analysis of retained items.

BASIC ANALYTICAL SCHEME (POWDERS, TAR, AND CHUNK SUBSTANCE)

The analyst must determine the appropriate sampling techniques, methods of recovery, extraction procedures, and methods of analysis to be used for the identification of a substance on a case-by-case basis.

• One confirmatory instrumental test (either FTIR or GC/MS) and at least one other

positive test (including chemical screening tests, microcrystalline, TLC, UVNIR, GC/MS or FTIR) is required for identification of an unknown substance. The combination of tests chosen must identify the specific substance present and must eliminate the possibility of a false positive identification.

REFERENCES:

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- If a case contains multiple containers of powder, tar, or chunk substance that are similar in appearance (appearance refers to the actual powder, etc., not similar packaging), then sufficient containers will be sampled to ensure that the highest weight limit is surpassed. Containers which are not sampled should be documented and reported as retained with no analysis.
- A weight is determined and recorded for all powders, tar, and chunk substance to be reported. If the weight is at a cut-off weight (i.e., 1.0 grams, 4.0 grams etc.), then the next significant figure other than zero is recorded and reported. The balance used to determine the weight shall be indicated on the examination sheet. It is the analyst's responsibility to verify that the balance used conforms to the laboratory's calibration guidelines.

Data required for instrumental analyses

The data generated from an instrumental method is documented with the unique case identifier and item designators listed's handwritten initials on every page. The date that the data based must be recorded on the examination sheet. The following should be documented:

UV: All appropriate information regarding sample preparation, wavelengths, weights, absorbances, or calculations could be documented on the UV graph or in the notes.

GC/MS: All appropriate information regarding retention times and library matches should be documented on the GC/MS graph(s) or in the notes. A graph of the blank run prior to the sample should be maintained with the case file.

; information regarding sample preparation and

should be documented on the FTIR graph or in the notes.

Maintenance and quality assurance procedures are documented and available by each instrument. It is the analyst's responsibility to verify that an instrument is working properly before use.

Non-instrumental methods may be used to aid in the analysis of powders, tar, and chunk substance. These methods may include thin layer chromatography, microcrystalline tests, and chemical screening tests:

(1 } Thin Layer Chromatography

Each solvent system used is listed on the examination sheet. The

observations are documented as well as the standards used for comparison.

REFERENCES:

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(2) Microcrystalline Tests

Each reagent system used is listed on the examination sheet. The observations are documented. This documentation may be either a written description of the microcrystal or a drawing. In addition the performance of blank controls is documented on the examination sheet.

(3) Chemical Screening Tests

Any reaction observed by the analyst is documented on the examination sheet by writing the color observed. In addition the performance of blank controls and spot plate checks are documented on the examination sheet.

If a quantitation is performed to determine the purity of an identified substance, then all appropriate documentation should be maintained with the case file.

LIQUIDS

- If a case contains multiple containers of liquid that are similar in appearance, then sufficient containers will be sampled so that the highest weight limit is surpassed. Containers which are not used should be documented and reported as retained with no analysis.

- A weight and volume should be reported on all liquids except those in an unusable container. In those cases, an estimated volume may be recorded.

It is common for volatile (PCP) liquids to evaporate rapidly (ether based solvent) so they should be analyzed on a priority basis. Because of this the amount obtained by the analyst may be less than the weight listed by the officer.

- For remaining analysis follow the analytical scheme given under powders, tar, and chunk substance.

TABLETS AND CAPSULES - GENERAL

- Tablets and capsules are generally identified as pharmaceutical or clandestine products. Pharmaceutical products are those manufactured by legitimate pharmaceutical companies who mark their products with logos which identify both the manufacturer and composition. Clandestine products by contrast are manufactured illegally and may have markings which simulate legitimate products,

but usually they are distinctive logos that represent commercial products, sports teams, or cartoon characters.

REFERENCES:

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~ ANALYSIS GUIDELINES CS-SOP 03 Page 5 of 11 --

- Tablets and capsules can typically be grouped based upon their appearance (size, color, and markings). Once separated into these groupings, each tablet and capsule should be considered an individual item for the purposes of sampling.
- For tablets and capsules that require analysis, follow the analytical schemes below based upon whether they can be identified as a pharmaceutical product or not. The combination of tests chosen must identify the specific drug present and must eliminate the possibility of a false positive identification. Tablets and/or capsules which are not analyzed should be documented and reported as retained with no analysis. ~

- A weight and number should be determined and recorded for all controlled substance tablets or capsules that will be reported. If a drug tablet or capsule is reported, no weight is necessary. If the number of tablets in one grouping is too numerous to count, then an approximate number is determined and noted. It is acceptable to describe the number as numerous instead of approximate on the supplement report. ~1

PHARMACEUTICAL TABLETS AND CAPSULES

- The first step in attempting to identify tablets and/or capsules is to compare their markings (logo) with reference texts. If they are successfully identified as pharmaceutical products, this is considered to be an acceptable screening test. When performing a pharmaceutical identification, a hardcopy (e.g. computer printout or xerox copy) will be included in the case file as well as documentation of the source. The logos observed by the analyst should be noted on the examination sheet. All attempts at identification, even those that are unsuccessful, should be documented on the examination sheet.

Some pharmaceutical products may not be identifiable by their logos as in the case of new products which published references are not available. In this case follow the analytical scheme for Clandestine Tablets and Capsules.

While partial logos can give useful information as to the possible identity of a pharmaceutical product, they cannot be used as a test for identification. Noting the results of partial logo searches on the examination sheet is acceptable as long as this is not used as a test. In this case follow the analytical scheme for Clandestine Tablets and Capsules.

When pharmaceutical identification is successful, only one tablet or capsule from each grouping needs to be fully analyzed by performing a confirmatory test (GC/MS or FTIR).

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if any analytical testing procedures indicate that tablets or capsules may be illicit, then pharmaceutical identification is no longer an acceptable test and the analytical scheme for Clandestine Tablets and Capsules should be followed.

CLANDESTINE TABLETS AND CAPSULES

- As a result of their clandestine origin, the actual composition of these tablets and capsules can vary greatly from item to item and appearance is generally useful only in grouping the items and is not an acceptable test for identification. The analytical scheme given under powders, tar, and chunk substance should be followed.

- For clandestinely manufactured tablets or capsules, a sample should be taken for the appropriate weight limit (based on the identified and tested charge). Tablets (capsules) which are not separately documented and reported as retained with no analysis.

For each grouping of tablets (capsules) to be sampled, each item up to 29 should be sampled for individual screening and a positive taken for GC/MS. For groupings with 30 or more tablets (capsules) at the analyst's discretion as to whether or not to sample more than 29 items for individual screening and a composite GC/MS.

For large numbers of clandestine tablets or capsules an alternative sampling plan which may be used is to take random samples to prove a statistically significant number of the items are in fact a controlled substance or dangerous drug. One method of random sampling lies on the theory of hypergeometric distribution. For a population (e.g. 100 or more tablets), randomly selecting 29 tablets from the population will be statistically conclude with a 95% probability that 90% of the population contain the substance identified in the selected random sample. To use statistics to make conclusions regarding a population the analyst should perform following steps:

1. Determine the total number of items in the population (grouping) to be sampled and record the total weight of the population.
 2. After selection each item is to be analyzed separately and completely.
 3. If presumptive testing indicates a difference in the randomly selected items, then all items in the population (grouping) will need to be analyzed separately or the population will need to be subdivided into separate groups as appropriate.
 4. Documentation should be noted on the examination sheet that statistical sampling was used.
- If the analyst has any questions regarding the sampling or analysis of clandestine tablets (capsules) he/she should consult with the Lab Manager or designee.

RE~ERENCES:

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DRUG RESIDUES

- Residues are samples which are either too small to be weighed accurately or that which remains after the bulk has been removed. Residues can be sampled by mechanical means (e.g. shaking or scraping) or chemical means (e.g. rinsing with solvent). Residues which are not sampled should be documented and reported as retained with no analysis.

A small amount of the residue is removed for analysis, ensuring that enough residue remains for an independent analysis. If the amount of residue is too small to retain a sufficient sample for reanalysis, then procedure blanks should be

used for that glassware, performed for the tests conducted. Procedure blanks for solvents, reagents, and instruments are completed prior to analysis of these samples. Documentation of procedure blanks is included in the case notes. Any procedure blank vials and/or sample extractions remain following analysis should be equilibrated to dryness, labeled appropriately, and retained with the case evidence.

Instrumental and non-instrumental methods may be used to aid in the analysis of residues (follow the analytical method even undetectable powders, tar, and chunk substance).

Visual examination of evidence is needed for charges (e.g. one suspect has a pipe and another uses only a push rod) indicates that no sample/residue is present for analysis (the push rod, then the item should be examined by a forensic analyst to confirm the lack of sample. Both analysts should initiate the notation on the examination sheet. The item is to be reported as analysis performed (no visible sample).

When field testers are received without any other evidence to analyze, they should be reported as "No unprocessed sample available for analysis." If requested to analyze the field tester, then the analyst should document the requestor information (name, phone number, and position) and handle appropriately depending on the amount of sample present for analysis (i.e. is there enough sample available for reanalysis).

PLANT SUBSTANCE AND PLANT SUBSTANCE RESIDUES

Samples should be taken from each container (bag, cigar, cigarette, etc.) of plant substance analyzed. If there are multiple containers, then sufficient containers will be sampled to ensure that the highest weight limit is surpassed. If the containers are cigarettes or cigars, then a sample should be taken from the middle of the item.

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ANALYSIS GUIDE UNCS-SOP 03 Page 8 of 11

Containers which are not sampled should be documented and reported as retained with no analysis.

•

Live plants:

7.

Plants are dried before weighing and analyzing.

2.

Remove roots, dirt and mature stalks before weighing (mature stalks = thick stalk ~1 centimeter or larger which test negative for THC content)

3.

The weight for the dried plants will be significantly less than the officer's listed weight.

.

A macroscopic and microscopic analysis is performed on samples taken. Any features found are documented on the Marijuana Check t. the identification of marijuana a minimum of 2 microscopic characteristics could be observed including cystolithic hairs or glandular hairs.

.

The Duquenois / Duquenois-Levine chemical test is performed if sufficient microscopic characteristics for identification of marijuana are observed.

.

Microscopic identification and at least one other positive test (including the Duquenois / Duquenois-Levine reagent test or GC/MS) are required for the identification of marijuana or drug seeds.

•

Germ test

Seeds for appearance. The viability of seeds may be determined by the seeds.

1. n.

2. Seed filter paper or the equivalent, and place in

3.

Incubate at room temperature for up to 14 days.

4. Document the number of seeds that germinated.

If any seeds germinate, it is determined that the seeds are capable of beginning germination.

For mushrooms or plant material suspected of containing psilocin / psilocybin a negative reaction in the Weber chemical screening test is sufficient to consider the sample negative for the presence of psilocin / psilocybin and the sample may be

reported as "No controlled substance identified". If the Weber test is positive a positive confirmatory test {GC/MS or FTIR} must be performed to report the presence of psilocybin.

REFERENCES:

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ANALYSIS GUIDELINES CS-SOP 03 Page 9 of 91 —

•

Instrumental and non-instrumental methods may be used when necessary as in the identification of THC in hashish samples (see the analytical scheme under powders, tar, and chunk substance).

. A weight is determined and recorded on all plant substance items that are analyzed including cigars, cigarettes, cigar stubs, and cigarette stubs. The weights determined for cigars and cigarettes should not include the weight of the wrapper (paper or tobacco leaf). At least one cigar or cigarette should be opened completely to determine the appropriate wrapper weight to subtract from the total sample weight. If cigar stubs and cigarette stubs need to be analyzed, the weight of the paper may be included in the total weight and this is indicated both on the report and on the examination sheet. If the weight of the cigarette stubs or cigar stubs makes a difference to the weight cut-off in the paper should be removed. Pipes and residues are not weighed. Marijuana weights are determined in metric units, they should be converted to ounces or pounds for the supplemental report.

. In cases where plant substance is combined with an identified controlled substance such as cocaine, phenylethylamine, codeine which cannot be easily separated from the plant substance, all weight is recorded in grams. For cigarettes or cigars dipped in resin or phenylethylamine liquid the entire weight is recorded (including wrapper paper / and the filter for manufactured items since it is contaminated with rolled substance). The plant substance from non-manufactured items in such cases should be tested to determine if it is marijuana. ~ ~

Analysis guidelines for cases can be found in the Disposed, Dismissed, and Destroy Case Guidelines.

Literature and Source Documentation

. R.S. Frank, et. al. "Representative Sampling of Drug Seizures in Multiple Containers," Journal of Forensic Sciences 36 (1991) pp: 350-357.

. SWGDRUG Recommendations, 2nd ed. "Part II A- Methods of Analysis/Sampling Seized Drugs for Qualitative Analysis", February, 2006.

. "Guidelines on Representative Drug Sampling", ENFSI, 2004. www.enfsi.org

REFERENCES:

SUBJECT/EVENT: PROCEDURE PAGE NUMBER:

NUMBER:

ANALYSIS GUIDELINES CS-SOP 03 Page 10 of 11
MODIFICATION SUMMARY
DATE VERSION CHANGE

02-01-09 2009 New format for Headers and Footers

p. 1- Change "... CS-SOP 01..." to "... Objectives Section..."
Second bullet point add "... then the residue should also be analyzed."

Powders, Tar, and Chunk Substance

Second bullet point remove "... aging), then-assess, sufficient..."

p. 3- Fifth paragraph required for instrumental analysis
delete "Refer to appropriate section in the training guide for future use."

p. 3- For Thin Layer Chromatography add "The observations are identical as well as the standards used for comparison"
Liquid
bullet point modify "... appearance, altered appearance"

..., when sufficient containers..."
Remove "An exception may be made ... , Consult with your supervisor if you have any questions."
Second bullet point change "... an estimated volume must be recorded."
Fourth bullet point delete "... and the Training Guide Section on Unknown Liquids."

Tablets and Capsules Section rewritten to separate into Pharmaceutical and Clandestine Sections. Include statistical sampling option for clandestine tablets and capsules

Drug Residues
Third bullet point remove "Refer to the Training Guide for additional information on the analysis of residues."

REFERENCES:

SUE3, IECT/EVENT: PROCEDURE PAGE NUMB~R~

NUMBER:

ANALYSIS GUI[3ELIPJES CS-SOP fl3 Page 11 of 11
Plant Substance and Plant Substance Residues
First bullet point modify "... plant substance analysed. #
~
... , then sufficient
containers..."

p. 7- Third bullet point modify "... the weight of the paper is may
be included in the total weight and this is to be
indicated..."

Fourth bullet point add "... which cannot be easily
separated from the substance, the total weight is..."

p. 7— Remove Destroy
Analysis by Analysts not in
Training} Section
Add referenced, Dismissed, and Destroy Case
Guidelines

Add 'r and Substantive Documentation
02-01-10

2010 Change 'r# as retained' to 'reported as retained with no
analysis throughout.

"ratory number" to "unique case identifier"
t u out. {per 09-3~-09 memo)

— Clandestine Tablets and Capsules section expanded to
include analyst's discretion as to whether or not to sample more
than 29 items for individual screening and composite GC/MS ~n
groupings of 30 or more items (third bullet point added).
per 06-09-09 memo)

p. 8-- Fifth bullet point "reported as No controlled substance" to
"reported as No controlled substance identified".

04-01-10

2Q10 p. 4(2) Microcrystalline Tests — Add "In addition the
performance of blank controls is documented on the examination
sheet."

p. 4(3) Chemical Screening Tests — Add "In addition the
performance of blank controls and spot plate checks are
documented on the examination sheet"

REFERENCES:

STANDARD OPERATING PROCEDURES

~

...w... . SUPPORT OPERATIONS
T*~ CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE ISSUED: I ~ EFFECTIVE DATE; PROCEDURE NUMBER

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SECTION: DATE OF REVISION: I REVISION NUMBER: PAGE NUMBER: ER:

02-01-10 S Page 1 of 4

SUBJECT/EVENT:

CASE DOCUMENTATION

SCOPE ~

These policies are established as minimum requirements for documentation and record keeping required for controlled substance cases.
CONTENTS OF CASE FOLDER

•

Report on the results of the analysis generated reports must contain the analyst's title and signature as written and signed.

•

Laboratory Evidence S~b ~orm and any other submission forms {e.g. Latent Lab, Property) r chain of custody records in printed or electronically retrievable form .

•

Laboratory exhibits with information about the exhibits contained in the evidence, tests performed with the appropriate observations, the results of any analyses, any other pertinent information. Each examination sheet must have a unique case identifier (HPD incident number or laboratory number) and item designators, the date for each observation and/or test, and the analyst's handwritten initials.

•

Analytical Data

1.

All charts, spectra, notes, and photographs will be maintained with the case file. Any photographs should be taped to or digital photos printed on 8 1/2" by 11" paper and labeled with the unique case identifier and item designators, the date the photos were taken, and the analyst's handwritten initials.

2.

All solvent blanks run prior to any case samples for the GC/MS should be maintained with the case file.

REFERENCES:

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CAS DOCUMENTATION CS-SOP Oa Page 2 of 4

- Any court orders or Motions for Discovery (labeled with the unique case identifier on each page and the initials of the analyst complying with the court order or Motion for Discovery).

- A record of all pertinent phone calls (labeled with the unique case identifier and initials).

Administrative Review

All case folders will be administratively reviewed prior to issuance of the report. An

administrative review should include the following:

- Verify that the incident number provided is the correct number for the case being entered. ~

- Verify all weights entered. It is very important to verify that the weights entered match the weights on the examination evidence since this information is used to charge the suspect. The weight on the submission form should also be checked to ensure that the analyst did not put the wrong designation, such as milligrams instead of grams.

- Verify all spelling, grammar, unique case identifier and item designators, and the analyst's name and file number. Results from all pages of the examination sheets should be included in the report.

- Verify that the information is listed for the evidence submitted.

- The completed administrative review is indicated at the end of the final 4LO generated with the abbreviation "AR" and the initials of the person entering the report. If the analyst enters his/her own report, "AR" is followed by the analyst's signature and the date signed.

Technical Review

All case folders will be technically reviewed. This review should include the following:

.
Verify that the weights on the report match the weights on the exam sheet.
Check that the weights on the submission form are consistent with the reported

weights.

. Verify that all spectra support the conclusion.

REFERENCES:

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NUMBER:

CASE DOCUMENTATION CS-SOP 04 Page 3 of 4

e Verify that all spectra contain the appropriate unique case identifier and item designators.

- Verify that all spectra contain any pertinent documentation and that the spectra are documented on the examination sheet. Check for the presence of any necessary blanks.

-

All examination sheets and spectra must have the analyst's handwritten initials.

-

Verify that all observations listed on the examination sheet are consistent with the conclusion(s).

-

The completed technical review is indicated on the) generated report with the abbreviation "TR" and the reviewer's initials reviewed.

Report Modification Records

It is sometimes necessary to modify a report after it has been issued. This may occur as the result of an Administrative Review, at the request of the DA's office, or for various other reasons.

When this occurs a supplemental report should be generated to explain the reason for the original report. This new supplemental report, and the modified report should all be retained in the case file.

REFERENCES:

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NUMBER:

CASE DOCUMENTATION CS-SOP 04 Page 4 of 4
MODIFICATION SUMMARY
DATE VERSION CHANGE
01-01-09 2009 New Format for Headers and Footers

p. ~- First bullet point add "...and signature as well as the date signed."

Analytical Data - Add "Any photographs should be taped to or digital photos recorded on 8 1/2" by 11" paper and labeled with the lab number the date the photos were taken, and the analyst's handwritten initials."

p. 2 - Administrative Review:

Fifth bullet point follows "The completed administrative review is attached at the end of the final report with abbreviation "AR" ... the analyst's signature the date signed."

p. 3 - Technical Review

Third bullet point modify as follows "The completed review is indicated on the final report with the abbreviation "TR" and the reviewer's initials and reviewed."

02-01-10 2009 Laboratory number" to "unique case identifier" output (per 09-30-09 memo)

'~ - First bullet point modify "Final report must contain..." to

"OLO generated reports must contain..."

p. 1- Second bullet point add "... or chain of custody records in printed or electronically retrievable format."

p.2 — Administrative Review:

Last bullet point add "...final OLO-generated report..."

p.3 -- Technical Review;

Last bullet point add "...final OLO-generated report..."

p. 3— Report Modification Records:

Second bullet point add "... a new supplemental OLO report..."

REFERENCE:

STANDARD OPERATING PROCEDURES
SUPPORT OPERATIONS
CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE ISSUED: EFFECTIVE DATE: PROCEDURE NUMBER

01-01-04 04-01-04 CS-SOP as

SECTION: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

04-01-01 017 Page 1 of 3

SUBJECT EVENT:

XAMINA710N SHEET

SCOPE

To provide guidelines for documentation of tests and on the examination sheet.

EXAMINATION SHEET

•

The first line is documented with the case identifier (HPD incident number or laboratory number), date, analyst's initials. The second line is documented with the item description to distinguish items within the case.

All observations are dated properly. For example, if a case is started and completed within the same day, then the date listed at the top is appropriate for all observations and tests. Any observations on a different date than the start date are documented accordingly.

Document all items appropriately in the space provided for "EVIDENCE SUBMITTAL". Notations and descriptions of evidence should be clear to a reviewer of the file. Extraneous items such as unused rolling papers, matches, loose pieces of paper (items that have no potential value for analysis and don't have visible residue) may be noted as visually negative but should not include a notation in the RESULTS section as they should not be reported.

•

When testing two or more items under one "test" (spot tests, microcrystalline, etc.), place the number of items and the observation.

•

When analyzing marijuana, the microscopic box, the Duquenois box and the Duquenois-Levine box are filled out with the number of samples tested and the observations. Pot may be used in the microscopic box to indicate that characteristics for marijuana were observed in the sample. These characteristics should be documented on the Marijuana Checklist.

REFERENCES:

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EXAMINATION SHEET CS-SOP 05 Page 2 of 4

Chemical screening tests (not tests) are documented by noting the observations and number of samples tested in the appropriate boxes.

- Spot plates are to be visually examined for cleanliness by the analyst prior to use. A check mark on the Examination Sheet next to "Spot Plate Check" indicates that the spot plates used were free of residue or debris.

-

The reagent system used for any microcrystalline tests is documented along with the observations.

-

Blank (or negative) controls for all chemical screening tests (including the Duquenois and Duquenois-Levine) as well as for all microcrystalline tests are performed at the same time as the sample testing. A check mark next to the tests performed indicates that no reaction was observed and that the blank control passed.

-

UV tests, GC/MS comparisons, and FTIR analyses are documented appropriately.

- The solvent system used to run chromatography plates is documented along with the observations as is standard(s) used for comparison. If UV visualization is used to visualize the plate this should be noted.

-

Information obtained from physical identifications (PHI) such as the DEA Logo Search, the PDR or the Desk Reference) or other references is recorded appropriately. Any successful pharmaceutical identification attempts are also recorded. Drawings (logos) observed by the analyst should be noted for comparison.

-

For items in which the entire submitted sample is used for analysis but a portion of the sample remains once the analysis is complete, the examination sheet should be documented appropriately.

For example: "All evidence used for analysis, remaining portion retained with the case."

For items where there is no sample remaining after analysis, the examination

sheet should be documented appropriately.

For example: "All evidence used for analysis, no remaining sample available." or use the abbreviation "EDIA" which stands for "Evidence Destroyed in Analysis".

Notations regarding the condition of the evidence when received should be

included on the Examination Sheet (e.g, moldy, wet, apparent blood) as well as

REFERENCES:

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EXAMINATION SHEET CS-SQP 05 Page 3 of 4

any procedures taken which may alter the appearance or weight of the evidence. Examples include removing needles from syringes, drying wet evidence (include length of time dried before weighing), drying of fresh plant material (include length of time dried before weighing) as well as removal of stalks, roots, and dirt. Measurable weights and volumes are recorded appropriately. The analyst will document on the examination sheet which balance is used for any weight determination.

When significant quantities of evidence are consumed during analysis, it is recommended that before and after analysis weights be noted on the exam

sheet. Alternatively, note the amount of sample used in analysis. The before analysis weight is to be reported on the supplement in cases. Examples include dilute cocaine liquids, large clandestine tablets and samples that are at a cut-off weight. ~ ,

The results of the analysis which " " are noted in the space provided for "RESULTS". If a quantity is done, the results of that analysis may be written under the name. If the results are negative, then "NCS" is written. If then note "Retain" in the space provided.

When a case is reopened later analysis is required, the following procedures should be followed: the original exam sheet is used:

1. The date of any additional testing is documented appropriately.

2. If the additional testing is performed by a different analyst, then his/her initials are documented.

Alternatively used following the proper guidelines for notations ~

REFERENCES:

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~ EXAMINATION SHEET CS-SOP 45 Page 4 of 4
MODIFICATION SUMMARY
DATE VERSION CHANGE
02-01-09 2049 New format for Headers and Footers

Scope delete "A more detailed... Training Guide."

p. 1- Third bullet point replace "followed by" with "and"
Combine fifth and sixth bullet points into the following
"Spot tests, UV tests, GC S comparisons, and FTIR
comparisons are documented appropriately."

p. 2- Second bullet point add "...

to with the observations as

well as the standard comparison. If UV
visualization is used the TLC plate this should
be noted."

Fourth bullet "For items in which the entire
submitted sample..."

Add bullet point "When significant quantities of
evidence at a cut-off weight."

re-meeting

~ 0-01-09 Memo Examination Sheet revised to change "L" in heading to "CASE"
09-30-09 a Checklist revised to change "LAB NUMBER: L" to

LAB NUMBER:"

01-01-10

Meeting Information Sheet revised to add row labeled "ITEM"
01-0

02-01-10 ~ 10 p. 1- Third bullet point modify "The first line is documented with
the unique case identifier (HPD

incident number or laboratory number), date, and

analyst's initials. The second line is documented with

the item designators used to distinguish items within the
case." (per 09-30-09 memo and 01-01-10 revision)

04-01-10 2010 p. 2-- Separate chemical screening tests and instrumentation
bullet point.

Add bullet points to document spot plate check and
blank controls for chemical screening tests.

Examination Sheet revised to document Spot Plate Check and

Blank Control Checks
REFERENCES:

STANDARD OPERATING PROCEDURES

0 d ~ , , , , SUPPORT OPERATIONS

~ , , , , r ~ CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCE SECTION

CATEGORY: DATE ISSUED: | EFFECTIVE DATE: PROCEDURE NUMBER
01-D 1-04 | 02-0 ~ -10 CS-SOP 06

SECTION: DATE OF REVISION: ~ REVISION NUMBER: PAGE NUMBER:
02-01-10 8 Page 1 of 5

SUBJECT: EVENT:

INSTRUMENT PERFORMANCE AND MAINTENANCE SCOPE

To establish quality assurance guidelines for the maintenance, and repair of analytical instrumentation and balances.

General Requirements for Analytical Instrument

All instruments will be periodically monitored and their performance verified in accordance with the manufacturer's ratings and specifications and HPD laboratory policy. All instruments' performance will be re-verified if they are moved or if a major repair is performed. It is the responsibility to ensure that appropriate re-verification has been done following an instrument on casework samples.

Performance verification records be maintained for a minimum of two years.

UV-Vis Spectrophotometry

- Conduct performance verification check on UV-Vis instruments quarterly or as needed.

Check the wavelength accuracy using the two characteristic wavelengths of deuterium light at 486.0 nm and 656.1 nm. Follow the manufacturer's specifications for performing this check. The peak wavelength ranges should be between 485.7 nm - 486.3 nm and 655.8 nm - 656.4 nm respectively.

- Standards will also be used to verify that the instrument is performing as expected. To do this weigh three samples of one of the validated standards (currently methamphetamine, heroin, or cocaine) and perform a quantitation using the experimentally determined E value. The determined purity should be within 10% of the expected value.

. Determine if the instrument meets specifications. If it does not, then the

instrument should be taken out of service until the issue can be resolved.

REFERENCES:

SUBJECT/EVENT: PROCEDURE PAGE NUMBER:

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INSTRUMENT PERFORMANCE AND MAINTENANCE CS-SOP 08 Page 2 of 5

- Maintain a logbook with the results.

FTIR Spectrometer

•

Conduct a performance verification check on the FTIR quarterly or more often as needed.

•

One method is to use the OMNIC Val-Q software to check the performance of the instrument. The measurements made by Val-Q are designed to meet a subset of the specifications contained in ASTM Standard Practice E142-99 and utilize two polystyrene samples, one 0.5 mil thick and one 3.0 mil thick. Val-Q tests the spectrometer's single-beam energy ratio, 10Q°/ peak-to-peak noise, 100% line root-mean-square (rms) noise, 1.5 mil polystyrene band position, resolution factor, and 3.0 mil polystyrene zeros. Field limits for each test are coded into Val-Q so that pass-fail results are reported.

- Maintain a logbook with the results.

Gas Chromatography/Mass Spectrometry

Performance Verification Check

•

The Mass Selective Detector should be tuned weekly when in use or more often as needed.

~_

•

The instrument should be maintained according to the manufacturer's instructions and must meet the manufacturer's specification.

•

A standard should be run daily when in use and the scan results entered in the logbook and aligned with the tune report for that week. If there is any deviation of standard m/z ratios, the instrument will be tuned and the standard re-run.

- Maintain a logbook with the results.

Other GC/MS Maintenance

•

Run a solvent blank before each sample run and maintain a copy of the blank run with the case file.

•

Perform regular and preventive maintenance according to the manufacturer's recommendations. A logbook documenting all non-routine maintenance (e.g.

REFERENCES:

S116J~CT1E1/ENT: PROCEDURE PAGE NUMBER: ,

NUMBER:

INSTRUMENT PERFORMANCE AND MAINTENANCE CS-SOP 06 Page 3 of 5
column~ replacement, filament replacement, seal replacement, vacuum oil~
changes, source cleaning, and major repairs) will be kept with the instrument.

Balances

- Laboratory personnel will check balances for accuracy regularly, using standard weights. Balances must be checked whenever they are moved from one location to another. Laboratory standard weights should be checked after the annual recertification of the balance.

-

Balances should be certified by an external vendor at least once a year.

-

Inspect the balances for cleanliness and check the level regularly.

-

The appropriate balance will be used for weight being measured and precision required. Care should be taken not to load a balance with too much weight.

-

Since the tolerances of electronic balances, the instrument specifications must be checked to determine appropriate criteria for satisfactory performance.

-

The following general specification may be used:

Balance class Weight Significant figures Acceptable variation

Analytical 4. 4.0000 g to 0.0001 g

1.0 1.0000 g \pm 0.0001 g

0.1 mg 0.0050 g to 0.0001 g

Top Loading 2.0 kg 2000.0 g to 0.1 g

1.0 kg 1000.0 g to 0.1 g

4.0 g 4.0 g to 0.1 g

1.0 g 1.0 g \pm 0.1 g

Back Scale 4.0 kg 4.04 kg to 0.02 kg

2.0 kg 2.00 kg to 0.02 kg

Other standardized weights may be used at the analyst's discretion.

• Analytical balances should be checked with standard weights at least weekly.

REFERENCE:

-

SUBJECT/E1/ENT:

INSTRUMENT PERFORMANCE AND MAINTENANCE

~ Top loading balances should be checked with standard weights monthly or as needed.

- The bulk scales should be checked with standard weights prior to use.

• Maintain a logbook with the results of the balance checks, standard weight checks, maintenance, and certification.

- It is the analyst's responsibility to verify that the necessary checks have been performed in the recommended time period for any balances or standard weights used.

Malfunction of an instrument or Balance

• If an instrument or balance fails the problem is detected during routine maintenance, the section manager or designee recorded in the logbook.

• No instrument or balance is to be ~

• Repair or have the instrument control procedures with standard instrument or balance ~ performed after routine maintenance be affected.

- Keep a record

Refrigeration

• Refrigerators are used for the storage of heat sensitive chemicals, standards, and reagents. They should be monitored at least once a week to ensure that they are working properly and within 2°C to 8°C. A record is to be maintained documenting the date checked, the displayed temperature, and the initials of the individual performing the check.

• If the temperature should fall outside of the acceptable range, verify that the unit has power and that air circulation has not been impeded. If corrective action does not return the unit to normal operation, then notify the section manager or designee. A technical representative may need to be called for service or the refrigerator may need to be replaced.

REFERENCES:

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NUMBER:

INSTRUMENT PERFORMANCE AND MAINTENANCE CS-50P 06 Page 5 of 5
MODIFICATION SUMMARY
DATE VERSION CHANGE
01-01-09 2009 New format for Headers and Footers

p. 2— GCIMS Performance Verification Check:
First bullet point change "...tuned weekly before use..." to
"...tuned weekly when in use..."
Third bullet point add "A standard should be run daily
when in use and the scan..."

p. 4 — Balances:
Second bullet point "All scales should..."

p. 4 — Malfunction of ,
not or Balance:
First bullet point "The supervisor or drug section
supervisor..." or "The manager or designee..."
02-01-10 2010 p. 4-- Balance

In maintaining a logbook for standard weight checks
Remember it is the analyst's responsibility to verify that the

All necessary checks have been performed
in the recommended time period for any balances or
standard weights used.

REFERENCES:

STANDARD OPERATING PROCEDURES

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1. SUPPORT OPERATIONS
CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE ISSUED: EFFECTIVE DATE: PROCEDURE NUMBER

01-01-04 02-01-10 CS-SOP 07

SECTION: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

02-01-10 6 Page 1 of 2

SUBJECT/EVENT:
GAS CHROMATOGRAPHY (GC) ~

This Section is rescinded as of June 1, 2004. ~
REFERENCES:

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SUSJECT/EV~NT: PROCED1R~ PAGE NUMBER:
NliMB~R:
GAS CHROMATOGRAPHY (GC) CS-SOP 07 Page 2 of 2
MODIFICATION SUMMARY

DATE VERSI~N CHANGE
01-01-09 2009 New format for Headers and Footers.
02-p~-10 2010 No Changes

R~F~RENCES:

0 STANDARD OPERATING PROCEDURES
SUPPORT OPERATIONS
ALABAMA CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION
CATEGORY: DATUM ISSUED: EFFECTIVE DATE: PROCEDURE NUMBER

01-01-04
02-a--o CS-SOP 8

SECTION:
DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

02-01-10 S Page 6 of 6
suai-cTrEVErrr:
GAS CHROMATOGRAPHY 1 MASS SPECTROMETRY (GC/MS)

scope An
analytical technique for the characterization of suspected
controlled substances, dangerous drugs and other substances.
SAFETY

- Use appropriate safety equipment wearing reagents and handling volatile
chemicals. Refer to the MSD data sheet for specific
chemicals.
- Properly secure high-pressure cylinders
- Use caution around hot surfaces such as oven interiors and injection and
detector ports
- Discard ampoules and any other pertinent materials in an appropriate
manner.

EQUIPMENT, MATERIALS, AND REAGENTS

- Gas chromatograph/mass spectrometer analytical instrument
- Auto-sampler vials and caps
- Solvent(s) appropriate for the substance being analyzed
- Microliter syringe (where applicable)

REFERENCES:

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SUBJECTIVE: PROCEDURE PAGE NUMBER:

NUMBER:

GAS CHROMATOGRAPHY 1 MASS CS-SOP 08 Page 2 of 6
SPECTROMETRY (GC/MS)

STANDARDS, CONTROLS, AND CALIBRATION

Calibration of the mass spectrometer is accomplished by tuning the instrument to ensure that the mass-to-charge ratios (m/z) are assigned correctly and to provide leak detection.

1. The instrument should be tuned weekly when in use according to the manufacturer's specifications and may be tuned more frequently as deemed necessary.

2. Tune records

are maintained in a file in the lab. If the tune is not successful, the instrument should be taken out of service until corrective action is taken. A

A standard should be injected daily to monitor performance when in use. The standard printout should be with the appropriate tune report. If the standard run does not provide a definite mass spectral identification, the instrument should be retuned and standard rerun. If the standard still does not provide an acceptable identification, then the instrument should be taken out of service until action is taken and the problem recorded in the logbook.

Solvent blanks are injected between case samples to verify that the column and system are free of contamination. The solvent blank should be run on the same method and immediately before it.

A procedure blank should be run for samples that will be completely consumed by analysis to verify the column, reagents, solvents, and laboratory glassware used are correct for the analysis of case samples. A procedure blank for GC/MS analysis should be prepared in exactly the same manner as the sample including the use of the same non-disposable glassware and solvents. The procedure blank is to be run on the GC/MS immediately prior to and using the same method as the sample run. Documentation of procedure blanks should be included in the case notes. If any sample remains after analysis, then the procedure blank vials and sample vials used should be evaporated to dryness, labeled appropriately, and retained with the case evidence.

Any significant peaks in the blank chromatograms should be properly investigated to identify their source (e.g. column breakdown, carryover from previous sample run, or instrumental contamination) so that corrective action can be taken as necessary. Any affected case samples and associated blanks

REFERENC~S:

' SUBJECT EVENT:

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SPECTROMETRY (GC/MS)

should be rerun (this is not necessary in the case of minor peaks identified as
column breakdown).

- For less frequently encountered controlled substances, standards should be run within the same timeframe that the evidence sample is tested, and a copy of the standard run should be retained in the case file. Examples of less frequently encountered substances include LSD, psilocin, or methaqualone. An acceptable timeframe for running the samples and standards would be within the same month as long as instrument conditions had not changed (column replacement or method modifications). Available and verified standards are a requirement for this practice.

PROCEDURE

GC/MS Operating Conditions

- Use appropriate temperature programs and other critical parameters to ensure that the suspected substance program should allow a reasonable time to elute.

- Lists of methods with standards available by each GC/MS system or are electronically retrievable. The lists provide guidance for those being analyzed. They are as needed for each sample preparation analysis.

Extract sample into a suitable solvent before they are injected into instrument. Print and retain the charts depicting the results of the GC/MS analysis in the case file. Include the following:

Take complete Total Ion Chromatogram (TIC) for each sample and corresponding blank run.

2. Mass spectra for all peaks corresponding to controlled substances and/or other substances of interest

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SPECTROMETRY (GC/MS)

3.

If a background subtraction is performed for a peak mass spectrum, then retain a copy of the original mass spectrum with the case file as well as the background subtracted mass spectrum. Note the retention time used to generate the background subtracted spectrum on the printout.

4.

Document the comparison of the unknown spectra to a known reference, either a stored library comparison or a literature reference. If a literature reference is used for comparison, cite the source.

5.

Each page will be printed, labeled with the unique case identifier and examiner's handwritten initials and will be maintained with the case file. Spectra or notes should have the item designation, test, and method of sample preparation (if not listed on the exhibit).

INTERPRETATION

•

Library searches can be used to provide information pertaining to the identity of a compound but should not be used as a replacement for verifying positive identification based on spectra.

•

If used for comparison, results from library searches must be printed and retained with the sample spectra.

LIMITATIONS

• When analysis is unable to provide positive identification, another

technique (Fractionation, etc.) must be utilized to provide positive identification.

• Some compounds may not be suitable for GC/MS analysis due to a variety of factors; for example, high injection port temperatures cause some compounds to break down or rearrange before they are ionized, preventing their identification. It may be difficult to identify individual compounds in a homologous series.

ADVANTAGES

•

Generally, mass spectra of compounds of interest are specific to single compounds and may be used for positive identification.

•

It may be possible to separate and identify complex mixtures that are difficult to separate through ordinary clean-up procedures.

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SPECTROMETRY (GC/MS)

- The technique is useful for analyzing small sample amounts that may be difficult to identify using other techniques.
A GC/MS auto-sampler increases the efficiency of analysis of numerous samples by functioning ~r~attended.

LITERATURE AND SUPPORTING DOCUMENTATION

- Douglas A. Skoog, Principles of Instrumental Analysis, 3rd Edition, (New York: Saunders College Publishing, { 985) 523-535, 554.
- F. W. Mc~afferty, Interpretation of Mass Spectra, 4th edition, (Sausalito, California: University Science Books, 1993).
- Jehuda Yiron, Forensic Mass Spectrometry, on, Florida: CRC Press, Inc., 1987).
- J. Throck Watson, Introductory Spectroscopy: Biomedical, Environmental, and Forensic Applications in New York: Raven Press Books, ~40 Avenue of the Americas, 1976}. _~\
- R. E. Andrey, "Mass Spectroscopy in Clarke's Isolation and Identification of Drugs, {London: The Pharmaceutical Press, 1981}, 251-263.

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REFERENCES:

SIJBJECT/EVENT: PROCEDURE PAGE NUMB~R~

NUMB~R:

GAS CHROMA70GRAPHY / MASS CS-SO~ 08 Page 6 of 6
SPEGTROMETRY {GC/IVfS)
M~DIFICATION SUMMARY
DATE VERSION CHANGE
Q1-01-09 2009 New forma# for Headers and Footers

p. 2— Standards, Con#rols, and Calibration:
First b~ilet point - 1. Added u...when in ~se..." and
remo~ed "... by the analyst andlor the laboratory
supervisor."

Second bullet point added ... en in use..."

Fourth bullet point mo ' sions about blank runs
to state that they o un on the same method
artd immediate sample run. Remove
example given er of procedure blanks and
sample runs If any sample remains after
analysis, ." and remo~ed "... in analysis..."

p. 3— First b

t ' t added "...controlled..." and remo~ed
opi MPP from lis# of less frequently
e un ed substances.

p. 4 a Preparation and Analysis:

emo~ed original sentence "Mass spectra for any

other pealcs..." and replaced with "If a bacfcground

subtraction is performed for a peak mass spectrum,

then retain a copy of the original mass spectrum with

the case ~le as well as the background subtracted

mass spectrum. Note the retention time used ta

generate the background subtracted spectrum on the

rintout.,,

02-41-10 2010 p. 4— Sample Preparatlon and Analysis:

5. Change "~aboratory case number" to "unique case
ident~er" (per 09-30-09 memo}. Change "exhibit
number" to "item desi r~ators".

REF~RENCES:

STANDARD OPERATING PROCEDURES

W. SUPPORT OPERATIONS
CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE ISSUED: EFFECTIVE DATE: PROCEDURE NUMBER

00-00-00 02-00-00 CS-SOS

SECTION: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

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SUBJECT/EVENT:

FURIER TRANSFORM INFRARED (FTIR) SPECTROMETRY

SCOPE

A non-destructive analytical technique used for the detection and identification of suspected controlled substances, dangerous drugs or substances.

SAFETY

Use appropriate safety equipment when using reagents. Refer to the MSDS for additional safety information for specific reagents.

EQUIPMENT, MATERIALS,

- Fourier transform infrared spectrometer
 - Mortar and pestle (if required)
 - Attenuated total reflectance (ATR) accessory
 - Acetone or suitable solvent (for cleaning)
- STANDARDS, CONTROLS, AND CALIBRATION

A performance verification check should be performed quarterly or more often as needed and recorded in an appropriate logbook. One method is to use the OMNIC Val-Q software to check the performance of the instrument. The measurements made by Val-Q are designed to meet a subset of the specifications contained in ASTM Standard Practice E1421-99 and utilize two polystyrene samples, one 0.5 mil thick and one 3.0 mil thick. Val-Q tests the spectrometer's single-beam energy ratio, 7.0% line peak-to-peak noise, 0.00% line root-mean-square (rms) noise, 1.5 micrometer polystyrene band position, resolution

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factor, and 3.0 mil polystyrene zeros. Factory-based limits for each test are coded into Val-Q so that pass-fail results can be reported.

- If the report obtained from a performance verification check indicates failure of one or more tests, consult the FT-IR Operation Troubleshooting section of the FT-IR Spectrometer Validation handbook for potential causes and corrective recommendations. If these do not correct the problem, the instrument should be taken out of service until corrective action is taken.

The test results obtained by utilizing the Val-Q performance checks are compared to prior results to verify that the system is working consistently over time.

A background should be taken before each : this step is included in the experimental method used for sample

PROCEDURE

Sample Preparation

. Use appropriate extraction necessary to isolate the sample. This may require : to a suitable salt form prior to analysis. ~

The sample must be in contact with the ATR accessory sampling area to provide the highest level of maximizing contact between the sample and sampling area in the following:

(1)

For liquid sampling, a trough insert is placed on the top of the ATR sampling area and fastened with the knurled mounting ring. The insert forms a shallow well around the ATR crystal face for containment of the liquid. For routine liquids, place a drop of sample in the trough insert and collect data. For volatile liquids, the volatile cover may be placed over the sample area to minimize evaporation of the sample.

(2) Solid samples may be placed directly onto the surface of the crystal (with or without the trough). Since the ATR effect only takes place very close to the surface of the crystal, an intimate contact has to be made by the sample on the ATR crystal surface. This is achieved by using the pressure clamp. With the sample in place over the crystal, lower the pressure tip by turning the control knob so that it is in contact with the sample. Continue lowering the tip until the clamp clutch clicks.

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Sample Analysis

~ Spectra are generally collected and printed with a resolution of at least 4 cm⁻¹

scanned from 4000 cm⁻¹ to 600 cm⁻¹ versus absorbance. This allows comparison to reference libraries with the same format. Spectral peaks should be of sufficient intensity to make an accurate comparison to known reference standards or published spectral data.

- Each spectrum will be printed, labeled with the unique case identifier and examiner's handwritten initials and will be maintained with the case file. Spectra or notes should have the item descriptors, date, and method of sample preparation (if not listed on the examination sheet).

- Document the comparison of the unknown spectrum to known reference and indicate the source of the reference in the file (issued or otherwise lab generated).

- If the subtraction function is used to remove interfering substances, then retain a copy of the original sample spectrum to use for future reference. Also note the substances subtracted to generate the result.

INTERPRETATION

- Library searches provide useful information pertaining to the identity but should not be used as a replacement for verifying positive spectral peaks.

- If used from library searches must be printed and retained.

- The infrared spectrum of the majority of controlled substances and other substances routinely identified is specific to a single compound and may be used for identification.

LIMITATIONS

- The sample must be relatively pure for positive identification.

- For an accurate comparison of an unknown spectrum to a standard spectrum, both samples (the sample and the reference) must be in the same salt form.

Some compounds may produce different crystal structures that can result in slightly different infrared spectra.

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- Infrared can usually be used to distinguish between optical isomers.

ADVANTAGES

-

Infrared is specific for the identification of controlled substances, dangerous drugs, and diluents and can be used as a confirmatory test.

-

Infrared is normally not a destructive test and the sample can be recovered for additional testing procedures, if necessary.

-

An unknown infrared spectrum can be quickly compared to known compounds found in drug libraries stored in the computer and then identified using published data from a reliable source or in-house spectra produced from known standards.

LITERATURE AND SUPPORTING DOCUMENTS

-

FT-IR Spectrometer Validation, Thermo Nicolet, Madison WI, 2001.

-

"Standard Practice for Describing Performance of Fourier Transform Mid-Infrared (FT-MIR) Centers: Level Zero and Level One Tests," ASTM E 1421-99, 1999.

-

Fell, A. F., Clarke's Illustrated Identification of Drugs, (London: The Pharmaceutical Society Britain, 1986).

-

Forensic Science, Volume I, ed. By Richard Saferstein, (Englewood Cliffs, N.J.: Prentice Hall, 1993).

-

Skoog, D. A., Principles of Instrumental Analysis, 3rd Edition, (New York: Saunders Co Publishing, 1985) 148-149.

REFERENCES:

SUBJECT/ENT: PROCEDURE PAGE NUMBER:

NUMBER:

' FTIR SPECTROMETRY CS-SOP 09 Page 5 of 5
MODIFICATION SUMMARY
DATE VERSION CHANGE
01-01-09 2009 New format for Headers and Footers

p. 3 - Sample Analysis:
Fourth bullet point added discussing subtraction
function and result in documentation needed
02-01-10 2010 p. 3— Sample Analysis:
Second bullet point changed to "unique case identifier"
to "unique case identifier" (see 30-09 memo).
Change exhibit number designators.

REFERENCES:

STANDARD OPERATING PROCEDURES

SUPPORT OPERATIONS

04

TOXIC LABORATORY DIVISION

CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE 15SEP: EFFECTIVE DATE: PROCEDURE NUMBER

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SECTION: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

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SUBJECT/CLIENT:

ULTRAVIOLET / VISIBLE SPECTROPHOTOMETRY (UVNIS)

SCOPE

A nondestructive technique for the preliminary identification of controlled substances, dangerous drugs and other substances. To establish a procedure to determine the concentration of a controlled substance, dangerous drug, or other substance in a sample using ultraviolet spectrophotometry. If UVNIS quantitation will be given after the general information section.

SAFETY

Use appropriate safety equipment when preparing reagents and pouring liquids. Refer to the MSDS for additional information for specific chemicals. Dispose of all chemicals in an appropriate manner.
EQUIPMENT, MATERIALS, REAGENTS

- UVNIS spectrophotometer

- Quartz cuvettes, matched pair, or equivalent
An appropriate solution for the sample

1. Acidic solutions, such as 2/3 N H₂SO₄

2. Basic solutions, such as 0.45 N NaOH

3. Methanol or ethanol
Analytical balance

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UVN1S SPECTROPHOTOMETRY CS-SOP 10 Page 2 of 7 STANDARDS, CONTROLS, AND CALIBRATION

- A UVN1S performance verification check should be performed quarterly or as needed and recorded in an appropriate logbook. Check the wavelength accuracy using the two characteristic wavelength peaks of deuterium light at 486.0 nm and 656.1 nm. Follow the manufacturer's specifications for performing this check. The peak wavelength ranges should be between 485.7 nm - 486.3 nm and 655.8 nm - 656.4 nm respectively.

- For comparison purposes, refer to reliable published reference materials, analyze known control samples or refer to in-house spectral sections produced from known samples.

- Reference solvent blanks should be run at the same time as the same solvent as sample.

- Refer to the end of this section for further information on UVN1S qualification.

- If an instrument fails a performance check or a performance problem is detected during routine maintenance or it could be taken out of service until corrective action is taken and then recorded in the logbook.

PROCEDURE

Spectrophotometer Operating Instructions

- The wavelength range for the UVN1S analysis of most drug samples is 340 to 220 nm, but should be expanded to accommodate certain substances such as Nitrofurantoin, GHB, and GBL.

Sample Preparation

- Dissolve the sample in a solution appropriate for the substance. Depending on the concentration of the sample, it may be necessary to dilute the solution so that the absorbance range is between 0.2 - 2 units. Plant materials will require extraction, while mixtures and other substances may require extraction prior to analysis.

Sample Analysis

- Collect a spectrum of the sample in the appropriate solution.

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- A "pH shift" may be performed on basic drugs in acidic solutions by adding an appropriate base until the solution is basic. For acidic drugs the QfOCe55 is reversed.

- Each spectrum will be printed, labeled with the unique case identifier and examiner's handwritten initials and will be maintained with the case file. Spectra or notes should have the item designators, date, and method of sample preparation (if not listed on the examination sheet).

Interpretation

The spectra obtained are evaluated with reference to document sources or spectra from known samples. The interpretation of spectra may be effected directly on the spectrum and should be documented on the examination in the appropriate category.

Limitations

- An ultraviolet spectrum is not sufficient if a positive identification cannot be made exclusively on the basis of UVA analysis.

- Not all substances absorb ultraviolet light; therefore the lack of absorbance or a flat-line spectrum is not necessarily an indication that a sample does not contain a controlled substance or drug (e.g. the dangerous drug carisoprodol has no UV absorption).

- The absorbance of a substance at any given wavelength may be modified by the presence of other compounds that also absorb at that wavelength. Additional sample preparation may be required to remove interfering compounds.

Advantages

- The test is quick and easy to perform.

- Usually very little sample preparation is required.

- UVNIS analysis is a good screening tool and routine analysis may provide information regarding the general concentration of the sample (strong, average or weak) and the presence or absence of some diluents (dilutants) and adulterants.

- This is usually a non-destructive technique and the sample can be recovered for other testing procedures, if necessary.

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IJVN15 SPECTROPHOTOMETRY CS-SOP 10 Page 4 of 7

May provide a quick and easy quantitation of some drugs/dilutants/adulterants.

QUANTITATION BY UV-NIS

Standards, Controls and Calibrations

- Performance verification check done quarterly or as needed. Standards will be used to verify that the instrument is performing as expected. To do this weigh three samples of one of the validated standards (currently methamphetamine, heroin, or cocaine) and perform a quantitation using the experimentally determined E value. The determined purity should be within 10% of the expected value. ~

- Reference solvent blank. ~

E₁, ^{cm} (E-Value) for the compound of this may be obtained from reference literature or determined with laboratory standards on the instrument prior to using this technique. If the latter procedure is used, the results will be documented in the appropriate logbook.

- Controlled substance reference used for the drug to be quantitated. ~

- If an instrument fails a pre-check or a performance problem is detected during routine maintenance or use, it should be taken out of service until corrective action is taken. The problem recorded in the logbook.

PROCEDURE

Sample Preparation

Obtain a representative sample of the substance requiring quantitation. The amount needed will vary according to the concentration of the controlled substance in the sample and the E-value or absorptivity of the controlled substance. For best results:

Adjust the concentration of the controlled substance so that the absorbance is strong enough to differentiate the peaks from background noise and yet weak enough to remain in the linear absorbance range.

2.

The sample must be diluted such that the absorbance is within the determined linear range.

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-

For powdered (solid) samples:

- 1.

To reduce the effects of the inherent percent error in weighing the sample on the final quantitation results, the analyst should use a larger quantity of the sample and dilute as necessary to obtain a solution that gives an absorbance in the linear range.

- 2.

Some samples may require extraction before they can be quantitated.

-

For liquid samples:

Liquid samples may vary greatly in concentration and should be extracted before quantitation, using an appropriate procedure for the substance being analyzed.

Sample Analysis

-

A baseline spectrum should be obtained over the desired wavelength range.

-

Collect a spectrum of the sample, recording the background at the appropriate wavelength for the drug being analyzed.

-

Calculations to determine concentration should be included in the case folder, either on the UV or the notes.

-

Each spectrum will be labeled with the unique case identifier and examiner's initials and will be maintained with the case file. Spectra or notes showing the instrument designators, date, and method of sample preparation will be noted on the examination sheet).
interpretation

The concentration will be calculated by application of the Beer-Lambert Law:

$A = abc$, where

A = absorbance value

a = path length [b = path length = 1 cm]

c = concentration

-

Note that the E-values in Clarke's are at 1.0% and must be divided by 10 in order

for the resultant calculation to yield a concentration (c) value of mg/ml (0.1 %).

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- For basic drugs, report the quantitation results in base form. The concentration as the salt may be reported only if the analyst has identified the salt form by an accepted analytical procedure.

Limitations

- UV quantitation is not suitable for samples that do not absorb UV light or for those that contain interfering compounds (such as nicotinic acid and pseudoephedrine in methamphetamine samples) that modify the absorbance of the sample at the quantitation wavelength.

- This technique is usually not suitable for samples with than one controlled substance.

A

- Samples that are not suitable for ultraviolet quantitation may be quantitated using an alternate technique such as gas chromatography.

Advantages

When analyzing relatively pure compound, it is quick and easy to perform, and requires less time and sample preparation than quantitation using gas chromatography.

LITERATURE AND SUPPORT INFORMATION

- Sandor Gorog, Ultraviolet-Visible Spectrophotometry in Pharmaceutical Analysis (CRC Press, 1995)

- A. F. Felton, "Ultraviolet, Visible, and Fluorescence Spectrophotometry", Clarke's Isolation and Identification of Drugs, Second Edition, (London: Taylor & Francis, 1986), 221-236.

- A.C. Moffat, et. al., "Ultraviolet, Visible, and Fluorescence Spectrophotometry", Clarke's Analysis of Drugs and Poisons, Third Edition, (London: The Pharmaceutical Press, 2004), 313-327.

- Douglas A. Skoog and Donald M. West, Principles of Instrumental Analysis (New York: Holt, Rinehart, and Winston, Inc., 1971).

- Terry Mills and Conrad J. Roberson, Instrumental Data for Drug Analysis, {New York: Elsevier Science Publishing Co., Inc., 1987}.

REFERENCES:

SUBJECT/EVENT; PROCEDURE RAGE NUMBER

NUMBER:

UVNIR SPECTROPHOTOMETRY CS-SOP 10 Page 7 of 7
MODIFICATION SUMMARY

DATE VERSION CHANGE

02-01-09 2009 New format for Headers and Footers

p. 1- Scope delete "...and in the Controlled Substances Training Guide."

p. 2— Standards, Controls, and Calibration:
Third bullet point change running of solvent blanks as deemed necessary by the analyst to running them at the same time with the same set as samples

p. 3 — Interpretation
Second sentence a "...on the spectrum of artifact should be documented examination sheet..."

p. 5 — Quantitation

S

Sample size

First sentence remove "...wavelength range 1.5-2.5

"Add reference to Clarke's 2004 Third Edition

02-01-10

2010 Update Analysis:

Second bullet point change "laboratory case number" to "unique case identifier" (per 09-30-09 memo). Change "exhibit number" to "item designators".

p. 5 — Sample Analysis:
Fourth bullet point change "laboratory case number" to "unique case identifier" (per 09-30-09 memo). Change "exhibit number" to "item designators".

REFERENCES:

IN STANDARD OPERATING PROCEDURE
O~
1. ~ SUPPLEMENTARY OPERATIONS
A'sa' ,,,, ^ ' ~ r CRIM ~ LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE 15/11/10: EFFECTIVE DATE PROCEDURE NUMBER

01-1-04 02-01-10 CS-SOP 11
sponsored: DATE OF REVISION: 1 REVISION NUMBER: PAGE NUMBER:
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SUBJECT/EVENT:
STANDARDS AND REFERENCES
SCOPE ~ ~

These policies serve to establish guidelines for the reference samples and
libraries.

1

Quality Control Procedures for Drug Standards

Before using a new drug standard or GC/MS will be performed to verify
that the compound is what it was intended to be. The spectra will be placed in a
quality control book which will contain all pertinent information such as the lot
number, source and initials of the analyst who performed the test.

« Some commercially drug standards are mailed with GC/MS and other
quality control data sheets will be retained.

Verification of Material Cannot Be Purchased Commercially

Thoroughly analyze and characterize any in-house samples before they are used
as a standard or reference.

- If a compound cannot be purchased and is obtained from another forensic
laboratory that has already encountered the problem or from a pharmacist (new
prescription drugs), however, the identity of the substance must be confirmed by FTIR
and/or GC/MS before it can be used as a reference. The verification data will be
retained in the laboratory.

Library References

When analyzing compounds, particularly drugs, using either GC/MS or FTIR, the
spectra will be compared to a reference standard. The source of the reference may be
an in-house library, a published library (such as NIST), any accepted published

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STANDARDS AND REFERENCES CS-SOP 1'~ Page 2 of 3

reference (such as Clarke's Isolation and Identification of Dn~gs), or a spectrum from a

reliable source (such as DEA). The instrument libraries on t~e FTiR include the

Georgia State Library and the in-hause library (HPD} generated from standards. The

instrument libraries on the GC/MS i~struments are the American Academy of Farensic

Science (AAFS) Library, the Na#iona! Institute of Standards and Technology (NIST)

~ibrary, and #he in-house libraries (HPD) generated f~om standards.

REFERENCES:

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STANDARDS AND REFERENCES C5-SOP 11 Page 3 of 3_
MODIFICATION SUMMARY

DATE VERSION CHANGE
01-01-09 2009 New format for Headers and Footers
02-01-10 2010 No Changes
REFERENCES:

STANDARD OPERATING PROCEDURES

~ ~ ~ ~ SUPPORT OPERATIONS
~ CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

CATEGORY:

DATE: ~ EFFECTIVE DATE: PROCEDURE NUMBER

01-01-04 ~ 04-09-10 CS-SOP 12

SECTION: pAT~ pF REVISION: 1 REVISION NUMBER: PAGE NUMBER:

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SUBJECT/EVENT:

REAGENT QUALITY ASSURANCE

SCOPE ~

The following describes quality assurance of reagents, chemical
reagents, and solvents used in drug analysis.

SAFETY

-

Use appropriate eye protection and safety equipment to avoid contact with
chemicals.

-

Refer to the appropriate MSD safe handling of chemicals.

-

Discard all chemicals and any other pertinent materials in an appropriate
manner.

PRACTICE

All pertinent reagent solutions will be labeled with the identity of the reagent and
the date of preparation (or lot number). A quality control logbook will be maintained and
will include the following information, when applicable:

- Reagent preparation date

- Preparer's initials

- Standard used and the results of a positive quality control check of the reagent

- Results of a negative (blank) quality control check of the reagent

- Initials of the analyst(s) who quality tested the reagent and the date of testing

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Quality Testing for Frequently Used Reagents

Frequently used reagents will be quality tested at the time of preparation and monthly thereafter. Upon preparation, the preparator will record his or her initials in the logbook along with the date prepared. This same date will also be reflected on the stock reagent container. The new reagent will be quality tested prior to being used and the appropriate information recorded in the logbook. The quality testing should include both a positive control using an appropriate standard and a negative (blank) control. In addition to the date of preparation, the date of the most recent quality test will be noted on the stock reagent bottle.

All general use containers (aliquots) of frequently used reagents will be quality tested monthly along with the stock reagent and the results recorded in the logbook. These containers will be labeled with the date of reagent preparation and the date of the most recent quality test. When a new stock reagent is placed in general use containers will be replaced with this reagent after it has been quality

Aliquots for reagents used as an analyst's reagent will be replaced each month from the stock reagent bottle after it has been rejected. These containers will be labeled with the date of reagent preparation and the date of the most recent quality test. It is the analyst's responsibility to document the use of his/her aliquots.

See the Chemical Screening Tests Sections for a listing of the current Frequently used reagents.

Quality Testing for All

Infrequently used reagents will be quality tested upon preparation and the results as well as the preparation date and the date of preparation will be recorded in the logbook. Subsequent quality testing will be performed by the analyst prior to use and the results as well as the standard will be documented in the case notes.

TLC (Thin layer chromatography) reagents will be quality tested during use by the analyst using an appropriate standard and the results will be documented in the case notes.

Upon preparation, acidic and basic solutions will be documented in the logbook with the date prepared, the preparator's initials, and the results of a pH check.

Quality Assurance

No reagent or other chemical preparation will be used in casework if it is not working properly or if it is contaminated.

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REAGENT QUALITY ASSURANCE CS-SOP 12 Page 3 of 4

If an analyst has reason to suspect that a reagent or other chemical preparation is not working properly or is contaminated, he or she must:

-

Check the reagent or system with standards or proper sample controls.

-

Discard the reagent if it fails the quality check, prepare a new reagent, and quality check the reagent with a known standard.

-

Cease performing casework with these reagents until the problem has been corrected.

-

Identify casework that may have been affected by the chemicals that failed the quality check and re-test with quality checks.

- Inform the Quality Manager if the problem persists.

Record

in logbooks or appropriate documentation.

REFERENCES:

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REAGENT QUALITY ASSURANCE CS-SOP 12 Page 4 of 4
MODIFICATION SUMMARY

DATE VERSION CHANGE

01-09-2009 New format for Headers and Footers

Quality Testing and Labeling of Frequently Used Reagents
sections both rewritten to remove the colored dot
procedure

Quality Testing of Infrequently used reagents by the
analyst modified to include noting test results and standard
used for testing in the case notes.

02-01-10 12Q10 No Changes

04-01-10 ~ 2010 p. 1 Practice — Delete 311 heading
Third bullet point added "Standard used and the
reagent .

Bullet point added "Results of a negative (blank)
quality control check of the reagent"

Point modified "Initials of the analyst who
labeled the reagent and the date of testing"
2 Quality Testing heading split into two headings:
Testing for Frequently Used Reagents and

Quality Testing for All Other Reagents

p. 2— Frequently used reagent section expanded to include
replacement of analysts in use aliquots each month and
the quality testing of general use aliquots each month.
Also include a negative (blank) control for monthly quality
checks.

REFERENCES:

STANDARD OPERATING PROCEDURES

1. SUPPORT OPERATIONS

1.1

CRIME LABORATORY DIVISION

CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE ISSUED: EFFECTIVE DATE PROCEDURE NUMBER

01-Q1-04 04-01-10 CS-SOP 13

SECTION: DATE OF REVISION: I REVISION NUMBER: PAGE NUMBER:

04-01-10 ~ 7 ~ Page 1 of 20

suave/ver:

CHEMICAL SCREENING TESTS

Scope

To describe the chemical screening procedures conducted as color tests or spot tests, for preliminary tests of controlled substances and non-controlled substances.

Safety

- Chemical spot tests may use a variety of corrosive, caustic, or other dangerous chemicals. Caution should be practiced, and appropriate personal protective safety equipment used.

Any solutions that fail a check should be discarded in an appropriate manner.

- Refer to MSD or other safety information for specific chemicals. Equipment, Materials, and Reagents

Spot plates, pipettes, or other appropriate containers/items.

- Reagents appropriate to the specific chemical spot tests.

Standards and Controls

- Each spot test stock reagent must be labeled with the name of the reagent or solution as well as the date of preparation (or lot number). A quality control log book will be maintained and will include the preparer's initials and the date prepared as well as the results of appropriate quality testing.

The frequently used spot test reagents are Ferricyanide, Marquis, Van Urk's, Cobalt thiocyanate, and Duquenois. These reagents will be quality tested at the REFERENCE:

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NUMBER:

CHEMICAL SCREENING 7~STS CS-SOP 13 Page 2 of 2Q

time of preparation and monthly thereafter with the date of preparation and most recent quality testing noted on all in-use containers. All other spot test reagents are considered infrequently used and must be quality checked at the time of preparation and prior to use. It is the responsibility of the analyst to quality check infrequently used reagents and document appropriately on the examination sheet. See the Reagent Quality Assurance Section for further explanation of quality testing procedures.

- It is the responsibility of the analyst to determine if reagents are working properly. Blank (or negative) controls for chemical screening tests are to be performed at the same time as sample testing to demonstrate that the reagents used are not contaminated. If the blank control shows a positive result (is not negative), then the reagents will be discarded and replaced with quality tested aliquots. In addition, spot plates used to perform screening tests are to be visually examined by the analyst prior to use that they are free of debris or residue. If a spot plate is not clean, it will not be used for analysis.

Definitions

Purified water means water that is purified by deionization or distillation. All water used to prepare spot test reagents will be reagent water.

Limitations

- All spot tests are presumptive and serve only as a guide for an analyst's analytical scheme.

- Adulterants and mixtures may produce reactions that interfere with the clear interpretation of results.

A sample with a low concentration of a particular substance may yield negative spot test results.

Advantages

Spot tests provide a quick and easy method for determining what type of compound or functional group a sample might contain.

- Spot tests can assist in the determination of appropriate analytical processing, collection of appropriate samples, and the grouping of samples for uniformity testing.

REFERENCES:

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CHEMICAL SCREENING TESTS CS-SOP 13 Page 3 of 20
Interpretation

-

Any reaction observed by the analyst will be documented on the examination sheet by writing the color observed.

- With weak color changes, the analyst may choose to document the color preceded by the designation "weak."

REFERENCES:

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NUMBER:

CHEM~CA~ SCREENING TESTS CS-SOP 13 Page 4 of 20

KOPPANYI TEST

Reagents\Chemicals

- Cobalt nitrate, $\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$

- Isopropylamine

- Methanol

1% Cobalt Nitrate Reagent#: Dissolve 8.0 g $\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$ in 500 ml methanol.

5% Isopropylamine Reagent Add 5 ml isopropylamine to 95 ml methanol.

(Reagent stored in the refrigerator).

Quality-test reagent with a barbiturate standard. ~~~

Procedure

1. Combine a small amount of sample and w d ps of 1% cobalt# nitrate reagent.

2. Record any observations.

3. Add a few drops 5% isopropylamine to sample.

4. Record any observations.

Interpretation

- Formation of a purple color upon addition of the 1% cobalt nitrate reagent indicates the possible presence of gamma-hydroxybutyrate (GHB).

- A few of will form a purple color with the addition of the first reagent.

- Formation of a purple color which forms after the addition of the 5% isopropylamine reagent indicates the possible presence of barbiturates.

- Sometimes vitamin C, ibuprofen, and lactose fillers in tablets will exhibit a faint purple color.

Literature and Supporting Documentation

- H.M. Stevens, 1986. "Color Tests" in Clarke's Identification and Identification of Drugs, ed. A.C. Moffat (London: The Pharmaceutical Press) pp. 128-147.
J. W., Jr. Stall, "The Cobalt Nitrate Color Test," Microgram 13(3), '98a, pp. 40-43.

REFERENCES:

SUBJECT/EVENT: PROCEDURE PAGE NUMB~R~
NUMBER:

CHEMICAL SCREENfNG T~57S CS-S~P 13 Page 5 of 20

• J.A. Morris, "Extraction af GHB for FTIR Analysis and a New Color Test for Gamma-Butyrolactone (GBL}," Microgram 32(S), 1999, pp. 215-221.
RE~ERENCES:

SUBJECT: PROCEDURE PAGE NUMBER:

NUMBER:

CHEMICAL SCREENING TESTS C5-SOP 13 Page 6 of 20
FERRICYANIDE TEST (also known as Simon's test)

Reagents/Chemicals

- Sodium nitroferricyanide (sodium nitroprusside)
 - Acetaldehyde
 - Purified water
 - 20% Sodium carbonate
- Ferricyanide Reagent: Dissolve 4 g sodium nitroferricyanide in a mixture of 40 ml acetaldehyde and 400 ml water. (Reagent stored in the refrigerator)
Quality-test reagent with a methamphetamine standard.
- Procedure

1. Combine a small amount of sample with a few drops of ferricyanide reagent.
 2. Add a few drops of 20% sodium carbonate
 3. Record any observations.
 4. The reagent combination itself turned red. This color is the normal color for a negative reaction.
- Interpretation

- Formation of a blue color for the addition of the 20% sodium carbonate indicates the presence of secondary amines (e.g. MDMA, methamphetamine, amphetamine, BZP, TFMPP).
 - Some secondary amines (MDE, N-OH MDA) do not form a blue color or form only a slight color due to steric hindrance.
 - Strongly basic solutions will form a deep red color before the addition of the 20% sodium carbonate.
- Literature and Supporting Documentation
- H.M. Stevens, 1981. "Colour Tests" in Clarke's Isolation and Identification of Drugs ed. A.C. Moffat (London: The Pharmaceutical Press) pp. 28-47.
- REFERENCE:

SUBJECT/EVENT: PROCEDURE PAGE NUMBER:

NUMBER:

CHEMICAL SCREENING TESTS CS-SOP 13 Page 7 of 20

MARQUIS TEST

Reagents/Chemicals

- Concentrated sulfuric acid (H₂SO₄)
 - Formaldehyde solution {~ 37% formaldehyde; stored in the refrigerator}
- Quality-test reagent with a standard of amphetamine, methamphetamine, or an opiate.
- Procedure

1. Combine a small amount of sample with a few drops of treated H₂SO₄.
2. Add one drop of formaldehyde solution.
3. Record any resulting color reactions.

Interpretation

- Formation of an orange to brown color indicates the possible presence of amphetamine, methamphetamine, or other substances may show similar color formations).
- Formation of a purple to black color indicates the possible presence of MDMA, MDE, and MDA.
- Formation of a green to black color indicates the possible presence of dextromethorphan.
- Formation of a color indicates the possible presence of heroin, other opiates, morphine, or quinine.
- Formation of a yellow color with the concentrated acid indicates the possible presence of diphenhydramine.
- Formation of a red color indicates the possible presence of salicylates (Aspirin).
- Formation of a black color upon the addition of the concentrated H₂SO₄ then orange with fizzing upon the addition of formaldehyde solution (due to the release of NO₂) indicates the possible presence of a nitrite.
- Formation of a dark red color indicates the possible presence of toluene.

REFERENCES:

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- A yellow powder which forms a deep p~rple color with the addition o# the concentrated HZSO4 followed by a change to yellow with t#~e addition af the formaldehy~e solution indicates the possible presence of tetracycline.

-

Some benzodiazepines such as diazepam form an orange color after se~eraf minutes.

- There may be other substances that form various colors wi#h the reagents.

Literature and Supporting Documentation

H.M. Stevens 1986. "Colour Tests" in Ciarke's I~

Dru~, ed. A.C. Moffat (London: The Pharmaceutical

S.H. Johns, et. al. "Spot Tests: A Color Cha~ Forensic Chemists,"

Journal of Forensic Sciences 24 {1979) pp. 63

REFERENCES:

' SUBJECT/EVENT: PROCEDURE PAGE NUMBER:

NUMBER:

CHEMICAL SCREENING TESTS CS-SOP ~3 Page 9 of 20

VAN URK'S TEST (also known as p-Dimethylaminobenzaldehyde or Ehrlich's Test)
Reagents/Chemicals

- p-Dimethylaminobenzaldehyde (p-DMAB)

- 95% Ethanol

Concentrated sulfuric acid

Van Urk's Reagent Dissolve 4 g p-DMAB in 450 ml 95% ethanol. Very slowly add 50 ml concentrated sulfuric acid (Reagent stored in the refrigerator)

Quality-test reagent with benzocaine, procaine, or lysergic acidamide.

Procedure ~

1.

Combine a small amount of sample and a few drops of Urk's reagent.

2. Record any observations. ~1

Interpretation

~ Formation of a bright yellow color is the possible presence of primary aromatic amines such as procaine and benzocaine.

-

Formation of a purple color indicates the possible presence of LSD and some other ergot alkaloids (reaction can take as long as five to ten minutes to occur).

Literature and Supporting Documentation

-

H.M. Steinen 6. "Colour Tests" in Clarke's Isolation and Identification of Drugs ed. A.C. Moffat (London: The Pharmaceutical Press) pp. 128-147.

-

S.H. Johns, et. al. "Spot Tests: A Color Chart Reference for Forensic Chemists," Journal of Forensic Sciences, 24 (1979): pp. 31-49.

Basic Training for Forensic Drug Chemists, U.S. Dept. of Justice, 3rd edition.

REFERENCES:

SUBJECT EVENT: PROCEDURE PAGE NUMBER:

NUMBER:

CHEMICAL SCREENING TESTS CS-SOP 13 Page 10 of 20

COBALT THIOCYANATE 1 MODIFIED COBALT THIOCYANATE TEST

(Cocaine test; Scott's test)

Reagents/Chemicals

- Cobalt thiocyanate
 - 95% USP glycerine
 - Purified water
 - Concentrated hydrochloric acid
 - Chloroform
- Cobalt thiocyanate Reagent: Dissolve 2 g cobalt thiocyanate in 100 ml water and dilute with 10 ml glycerine.
- Quality-test reagent with a cocaine standard.
- Procedure

1. Combine a small amount of sample with cobalt thiocyanate reagent.
2. If a color change is observed, record reaction.
3. Add one drop of concentrated HCl acid.
4. Add a few drops of chloroform to form any soluble complexes.
5. Record any observations.

Interpretation

If addition of cobalt thiocyanate reagent results in the formation of a blue color which dissolves upon addition of the concentrated HCl and reappears in

the chloroform layer, then a cocaine salt could be present.

If addition of the cobalt thiocyanate reagent results in no color formation or a light blue color around the surface of the particles followed by a blue color with addition of concentrated HCl which transfers to the chloroform layer, then cocaine base could be present.

The cobalt thiocyanate test is a useful step in distinguishing cocaine salt from cocaine base.

Some other substances that form a blue color with the addition of the cobalt thiocyanate reagent are acetone, lidocaine, PCP, heroin (if concentrated enough), gamma-butyrolactone, and diphenhydramine.

REFERENCES:

SUBJECT/EVENT:
PROCEDURE PAGE NUMBER:

NUMBER:

CHEMICAL SCREENING TESTS CS-SOP 13 Page 11 of 20
Literature and Supporting Documenta#ion

- - L.J. Scvtt, "Specific F'ield Test for Cocaine, " Microgram fi(1973}: pp. 179-181.
 -
 - H.M. Stevens, 1986. "Cofour Tests" in Clarke's Isolation and Identification of Druas• ed. A.C. Moffat (London: The Pharmaceutical Press) pp. ~28-147.
 -
 - A.L. Deakin, "A Study of Acids Used for the Acidified Cobalt Thiocyanate Test for Cocaine Base," Microgram Joumal1(1-2}, Jan-Jun~ 2003, pp. 40-43.
 -
 - S.H. Johns, et. af. "Spot Tests: A Color Chart Referenc r Forensic Chemists," Journal of Forensic Sciences 24 (1979) pp. 631-fi49.
 -
 - J.A. Morris, "Extraction of GHB for FTIR An ysi ew Color Test for Gamma-Butyrolactone (GBL}," Microgram 32 , . 215-221.
- REFERENCES:

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CHEMICAL SCREENING TESTS CS-SOP ~3 Page 12 of 20

JANOVSKY TEST

Reagents/Chemicals

-
- m-Dinitrobenzene
-
- 95% Ethanol
-
- Purified water
- Potassium hydroxide
- 2% m-Dinitrobenzene Reagent Dissolve 4 g m-dinitrobenzene in 200 ml 95% ethanol.
- 5 N Potassium Hydroxide: Dissolve 56 g potassium hydroxide in 100 ml water.
- Quality-test reagent with diazepam standard.

Procedure

1. Combine a small amount of sample with equal parts of 2% m-dinitrobenzene reagent and 5 N potassium hydroxide.

2. Record any observations.

Interpretation

- Formation of a purple color indicates the possible presence of diazepam or flunitrazepam.

• Same references indicate that ketamine will form a blue color with the test, but our observations have been that the color formation is to purple and not consistent enough for reliability.

• Formation of a yellow color indicates the possible presence of clonazepam or nitrazepam.

- No color formation is seen with alprazolam or lorazepam.

Literature and Supporting Documentation

• C.L. Rucker, "Chemical Screening and Identification Techniques for Flunitrazepam," Microgram 31(7), 1998, pp. 198-205.

REFERENCES:

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CHEMICAL SCREENING TESTS CS-SOP 13 Page 13 of 20
WEBER TEST

Reagents/Chemicals

-

Fast Blue B salt

-

Concentrated hydrochloric acid

-

Purified water

0.1 % Fast Blue B Reagent: Dissolve 0.1 g Fast Blue B salt in 100 ml water.
Prepare this reagent fresh and quality-test with standard psilocin before use.

Procedure

1.

Combine a small amount of sample or material from the mushroom sample
with a few drops of the 0.1 % Fast Blue B and wait approximately 1
minute.

2.

Record any observations.

3.

Add a few drops of concentrated hydrochloric acid.

4. Record any observations.

Interpretations

-

Formation of a red color indicates the presence of the Fast Blue B reagent which changes
to blue with the addition of acid indicates the possible presence of psilocin.

Literature and Supporting Documentation

-

A.S. Garr S.R. Lemons, J.H. Gaskill, "The Weber Test: A Color Test for the
Presence of Psilocin in Mushrooms", SWAFS Journal, 15(1), April ~ 1993, pp. 44-45.

REFERENCES:

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CHEMICAL SCREENING TESTS CS-SOP 13
FERRIC CHLORIDE TEST
Reagents/Chemicals

- Ferric chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$
 - Purified water
- 5% Ferric Chloride Reagent Dissolve 8.3 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 100 ml water.
Quality-test with gamma-hydroxybutyric acid (GHB) standard.

Procedure

1.
Combine a small amount of sample with a few drops of 5% reagent.

2. Record any observations.

Interpretation

- Formation of a red-orange color indicates possible presence of GHB.
- A

- Formation of a dark purple color indicates the possible presence of salicylates (aspirin).

• Formation of a blue color indicates the possible presence of acetaminophen.

Literature and Supporting Documentation

• H.M. Sieben, "Four Tests" in Clarke's Isolation and Identification of Drugs, ed.

A.C. Moffat (London: The Pharmaceutical Press) pp. 128-147.

• A. Morris, "Extraction of GHB for FTIR Analysis and a New Color Test for Gamma-Butyrolactone (GBL)," Microgram 32(8), 1999, pp. 215-221.

REFERENCES:

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CHEMICAL SCREENING TESTS CS-SOP 13 Page 15 of 20

LIEBERMANN TEST

Reagents/Chemicals

- Sodium nitrite

~ Concentrated sulfuric acid (H₂SO₄)

Liebermann's Reagent Carefully add 5 g sodium nitrite to 50 ml concentrated H₂SO₄ with cooling and swirling. Perform the addition in the hood, as toxic nitrogen

oxides are produced.

Quality-test the reagent with a standard of methamphetamine, amphetamine, mescaline, or dextropropoxyphene.

Procedure

1.

Combine a small amount of sample and a few Liebermann's reagent.

2. Record any observations.

Interpretation

•

Various colors may be formed by large number of different compounds.

Results or interpretations cited in Steens (1986).

•

A variety of color results may be found in Chiong (p.491).

V

Literature and Supporting Documentation

•

H.M. Steens, "Colour Tests" in Clarke's Isolation and Identification of Drugs, ed. A. at (London: The Pharmaceutical Press) pp. 127-147.

•

D.M. Chiong, E. Consuegra-Rodriguez, and J.R. Almirall, "The Analysis and Identification of Steroids", Journal of Forensic Sciences, 37(2), March 1992, pp 488-502.

REFERENCES:

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CHEMICAL SCREENING TESTS CS-SOP ~ 3 Page 16 of 20

SULFURIC ACID TEST

Reagents/Chemicals

- Concentrated sulfuric acid

Quality-test reagent with a steroid standard.

Procedure

1.

Combine a small amount of sample and a few drops concentrated sulfuric acid.

2. Record any observations. A UV light may be used at visualization of a color change.

Interpretation

. Formation of an orange or yellow color indicates the possible presence of a steroid.

Formation of a yellow color indicates the possible presence of diphenhydramine.

Literature and Supporting Documentation

. H.M. Stevens, "Tests" in Clarke's Isolation and Identification of Drugs, ed. A. Martin: The Pharmaceutical Press) pp. 127-147.

•

D.M. Chi, E. Zadríguez, and J.R. Almirall, "The Analysis and Identification of Drugs" of Forensic Sciences, 37(2), March 1992, pp 488-502.

REFERENCES:

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CHEMICAL SCREENING TESTS CS-SOP 13 Page 97 of 20

MANDELIN TEST

Reagents/Chemicals

-

Ammonium vanadate

-

Concentrated sulfuric acid

-

Purified water

Mandelin's Reagent: Dissolve 0.5 g ammonium vanadate in ~.5 ml water. Carefully dilute to 100 ml with concentrated sulfuric acid. Filter the reagent through glass wool.

Quality-test with a codeine standard.

Procedure

- 1.

Combine a small amount of sample and a few Mandelin's reagent.

2. Record any observations.

Interpretation

. Various colors may be produced by a large number of different compounds including codeine which is due to the formation of a green color. Results

and interpretations may be found in Stevens (1986).

. A variety of color changes for steroids may be found in Chiong (p. 491).

Literature and Supporting Documentation

-

H.M. Stevens: "Colour Tests" in Clarke's Isolation and Identification of Drugs, ed. A. C. Moffat (London: The Pharmaceutical Press) pp. 127-147.

-

D.M. Chiong, E. Consuegra-Rodriguez, and J.R. Almirall, "The Analysis and Identification of Steroids", Journal of Forensic Sciences, 37(2), March 1992, pp 488-502.

REFERENCES:

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CHEMICAL SCREENING TESTS CS-5~P 13 Page 18 of 20

DUQUENOIS 1 DUQUENOIS-LEVINE TEST

Reagents/Chemicals

- Vanillin
 - 95% Ethanol
 - Acetaldehyde
 - Concentrated hydrochloric acid
 - Chloroform
 - Petroleum ether
- Duquenois Reagent: Add 19.2 g vanillin and 2.4 ml acetaldehyde to 6 ml 95% ethanol. (Reagent stored in the refrigerator)
- Quality-test with a known marijuana sample.
- Procedure

1.
Place a small amount of plant material in a container.
2.
Either proceed to the next step or extract plant material with petroleum ether.
3.
If extracted, discard the plant material and evaporate to dryness.
4.
Add one part of the Duquenois reagent and wait approximately one minute.
5.
Add one part concentrated hydrochloric acid. (this is the Duquenois test)
6.
Record any observation
7.
Add one part chloroform (this is the Levine modification)
8. Record any observation

Interpretation

- Formation of a blue color after the addition of concentrated hydrochloric acid to the mixture of Duquenois reagent and plant material or extract is a positive reaction and indicates the possible presence of tetrahydrocannabinol (THC).
- Formation of a purple color in the chloroform layer indicates the possible presence of tetrahydrocannabinol.

Formation of a purple color in both reactions above indicates that the components (cannabinoids, including THC) unique to marijuana, marijuana residue, or hashish are present.

REFERENCES:

SUBJECT/EVENT: PROCEDURE PAGE NUMBER:
NUMBER:
CHEMICAL SCREENING TESTS CS-SOP ~3 Page 19 of 20
Literature and Supporting Documentation

•

C.G. Pitt, et. al. "The Specificity of the Duquenois Color Test for Marijuana and Hashish", Journal of Forensic Science, 17 (1972): pp. 693-700.

• K. Bailey, "The Value of the Duquenois Test for Cannabis — A Survey", Journal of Forensic Science, 24 (1979): pp. 817-841.

REFERENCES:

SUBJECT/EVENT: PROCEDURE PAGE NUMBER:

NUMBER:

CHEMICAL SCREENING TESTS CS-SOP ~3 Page 20 of 2a
MODIFICATION SUMMARY
DATE VERSION CHANGE
01-Q1-09 2009 New format for Headers and Footers

p. 1 and 2-"Color" test changed to "spot" test throughout
for consistency

p. 1- Standards and Controls:

Second and third bullet points revised and rewritten
to reflect changes in the labeling frequently used
reagent quality checks

For Ferricyanide Test add TFMPP to examples of
secondary amines indication of a blue color

For Marquis Test revision of a brownish color
indicates the presence of PCP liquid"

02-01-10 2010 No Changes

04-D1-10 2010 p. 1- Standards and Controls:

First bullet point add to last sentence "...and the date
recorded as well as the results of a representative
test in .

second bullet point add "...and monthly thereafter with
the date of preparation and most recent quality
testing..."

Third bullet point include blank controls and spot plate
checks.

REFERENCES:

STANDARD OPERATING PROCEDURE

0,

Support Operations

Metropolitan Crime Laboratory Division

CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE ISSUED: EFFECTIVE DATE: PROCEDURE NUMBER

04-01-90 CS-SOP 14

SECTION: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

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SUBJECT/EVENT:

MICROCRYSTALLINE TESTS

Scope

To describe procedures for the presumptive identification of controlled and noncontrolled substances using polarized-light microscopic crystalline reagents.

Safety 1

Microcrystalline tests may use a variety of, caustic, or other dangerous chemicals. Caution should always be and appropriate personal protective equipment used.

Refer to MSDS for additional action for specific chemicals and proper disposal.

Equipment, Materials

- Polarizing microscope with analyzer
- Glass slide including depression well slides
- Pipettes and graduated drapper bottles and other containers for the reagents
- Reagents appropriate to the specific microcrystalline tests.

Standards and Controls

- Each microcrystalline test stock reagent must be labeled with the name of the reagent or solution as well as the date of preparation (or lot number). A quality control log book will be maintained and will include the preparer's initials and the date prepared as well as the results of appropriate quality testing.
- The frequently used microcrystalline test reagents are aqueous Gold Chloride and aqueous Platinum Chloride. These reagents will be quality tested at the time of preparation and monthly thereafter with the date of preparation and most

REFERENCES:

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MICROCRYSTALLINE TESTS CS-SQP 34 Page 2 of 4

Recent quality testing noted on all in use containers. All other microcrystalline test reagents are considered infrequently used and must be quality checked at the time of preparation and prior to use. It is the responsibility of the analyst to quality check infrequently used reagents and document appropriately on the examination sheet. See the Reagent Quality Assurance Section for further explanation of quality testing procedures.

- It is the responsibility of the analyst to determine if reagents are working properly. Blank (or negative) controls for microcrystalline tests are to be performed at the same time as sample testing to demonstrate that the reagents used are not contaminated. If the blank control shows a positive reaction (is not negative), then the reagents will be discarded and replaced with fresh quality tested aliquots.

Limitations

The presence of other compounds, such as impurities, can inhibit the growth of the microcrystals and lead to deformation or lar shapes.

Advantages

- It requires very small amounts for a successful test.

- Most microcrystalline tests are relatively quick, easy, and specific for the compound tested.

Procedures

In general, the following are taken when analyzing case samples:

- Dissolve a small portion of the sample in a suitable solvent on a microscope slide.

- Place a small amount of reagent on a cover slip.

- Invert the cover slip and carefully drop onto the microscope slide allowing the reagent and sample solution to mix.

- Observe the formation of characteristic microcrystals under a microscope.

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MICROCRYSTALLWE TESTS CS-SOP 14 Page 3 of 4

Interpretation

Microscopic observations are documented on the examination sheet by writing a description or drawing of what is observed. .

Literature and Supporting Documenta#ion

- E.G.C. Clarke editor, Isolation and Identification of Drugs Volume 1, 1978.

"Microcrystal Tests", pp. 135-141.

REFERENCES:

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NUMBER:

MICROCRYSTALLINE TESTS CS-SOP 14 Page 4 of 4
MODIFICATION SUMMARY
DATE VERSION CHANGE
02-Q1-09 2009 New format for Headers and Footers

p. 1—"Standards, Controls and Calibration" changed to
"Standards and Controls"

Second and third bullet points combined and rewritten
to reflect changes in the labeling of frequently used
reagent quality checks

p. 2 — Advantages:

Remove first bullet point "Simple method for the
differentiation of oxidants."

p. 2— Procedures and

its title changed to

Procedures

General procedure added and statement "All
procedures in Training Guide." deleted

p. 3— Literature Supporting Documentation section
added

02-01-10 2014 No Change

Q4-01-10 2010 Standards and Controls:

First bullet point remove acidic Gold Chloride and
Potassium Permanganate from frequently used
microcrystalline test reagents

First bullet point add to last sentence "... and the date
prepared as well as the results of appropriate quality
testing."

Second bullet point add "...and monthly thereafter with
the date of preparation and most recent quality testing..."

Third bullet point include blank controls.

REFERENCES:

STANDARD OPERATING PROCEDURES

1. SUPPORT OPERATIONS
2. CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

CATEGORY:
or revision: EFFECTIVE DATE: PROCEDURE NUMBER

01-01-04 | 02-09 -1 a CS-SOP 15
SECTION: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

02-01-14 6 Page 1 of 5
SUBJECT: EVENT:
THIN LAYER CHROMATOGRAPHY (TLC)

SCOPE:
To describe the use of thin-layer chromatography as a method.
SAFETY

- Use appropriate eye protection, gloves to avoid any contact with the chemicals that are involved with this technique. This technique should be performed in a fume hood.
- Care should be used when preparing the TLC plates to avoid accidental ingestion of the reagent or exposure of skin and eyes to the reagent. Refer to the appropriate MSDS for safe handling of the solvents and reagents used in this technique.
- Developing solvent and indicator reagents should be discarded in an appropriate manner.

EQUIPMENT, MATERIALS, AND REAGENTS

- Silica gel on aluminum, glass, polyester, or other appropriate medium
 - Glass developing tank
 - Capillary tubes, micropipettes, or equivalent
 - UV light box (long and short wave)
 - TLC solvent systems and developing sprays as outlined in the Training Guide
- REFERENCES:

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THIN LAYER CHROMATOGRAPHY (TLC) CS-SOP 15 Page 2 of 5 STANDARDS AND CONTROLS

An appropriate known reference standard should be used to test the system and detection reagents. The standard should be analyzed with all case samples and a comparison of the Rf values documented.

PROCEDURE

In general, the following steps are taken when analyzing case samples:

- . Extract the sample with an appropriate solvent.
- . Spot a suitable amount of extract from the sample and the one standard on

the TLC plate approximately 1.5 cm above the bottom edge.

- . Allow the sample to dry after application.
- . Place the plate vertically into a solvent trough with enough solvent moisture to cover

0.5 to 1.0 cm of the sample end of the plate.

- . Allow the solvent front to rise near the top of the TLC plate.
- . Remove the plate from the trough and allow it to air dry. Systems containing ammonia may be gently removed the excess ammonia before spraying.
- . Apply an overspray and/or view under UV light to visualize the

components

- . Compare sample spot to that of the standard.
- . Document the solvent system used to analyze the samples and the results of

analysis noting the standards used for comparison.

INTERPRETATION

A positive determination is made when a spot(s) of an unknown substance matches the color and location of the standard.

LIMITATIONS

- . TLC is not considered a confirmatory test and further analysis is necessary for

the positive identification of a questioned substance.

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THIN LAYER CHROMATOGRAPHY (TLC) CS-SOP 15 Page 3 of 5

Various factors limit the determination of R_f values in TLC analysis, including the length of the plate, bleeding of the sample, temperature, and developing time. However, the use of multiple systems and chemical developing reagents make it a more specific technique.

ADVANTAGES

- TLC is a relatively quick and easy technique.
- It can be used as a clean-up procedure for complex mixtures.
- It requires no expensive instrumentation.

LITERATURE AND SUPPORTING DOCUMENTATION

• Bobbitt, J. M.; Schwarting, A. E.; Gritter, R. *Introduction to Chromatography*, 1968.

• A.C. Moffat, "Thin-Layer Chromatography in the Isolation and Identification of Drugs, 2nd edition (London: the Pharmaceutical Press, 1981), 910-177.

• Fox, R. H.; "Paper Chromatography", Isolation and Identification of Drugs, ed.

E.G.C. Clarke (London: The Chemical Press, 1969), 43-58.

• Miller, J. A.; Neuxil, E. *Organic Chemistry, Concepts and Applications*, (D.C. Heath & Company, Inc., Mass., 1979), 555.

• "Chromatography, Thin Layer Chromatography Tables, Volume I, Sec. II.IV"; *Handbook of Chromatography*, Volume I, edited by Robert C. Weast, CRC Press, Division of the Chemical Rubber Company, 1972, 477-487.

• "Practical Applications II.I Detection Reagents for Paper- and/or Thin Layer Chromatography", Volume 2, Section II, *CRC Handbook of Chromatography*, edited by Robert C. Weast, CRC Press, Division of the Chemical Rubber Company, 1972, 103-109.

• E. Buef, C. N. Plum, and S. K. Friesbie, "An Evaluation of a Partition Thin Layer Chromatography System for the Identification of Cannabinoids", *Microgram*, 15 (1982): 145-157.

REFERENCES:

SUBJECT: PROCEDURE: PAGE NUMBER:
NUMBER:

THIN LAYER CHROMATOGRAPHY (TLC) C5-SOP 15 Page 4 of 5

•

R.B. Hughes and R.R. Kessler, "Increased Safety and Specificity in the Thin-Layer Chromatographic Identification of Marijuana", Journal of Forensic Science, 24 (1979): 842-846.

•

R.B. Hughes and V.J. Wamer, Jr., "A Study of False Positives in the Chemical Identification of Marijuana", Journal of Forensic Science, 23 (1978): 304-310.

REFERENCES:

SUBJECT/EVENT: PROCEDURE PAGE NUMBER:

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THIN LAYER CHROMATOGRAPHY (TLC) CS-SOP 15 Page 5 of 5
MODIFICATION SUMMARY

DATE VERSION CHANGE

02-01-09 2009 New format for Headers and Footers

p. 2- Procedure ninth bullet point add "...the results of
analysis noted in the standards used for comparison."

p. 2- Procedure last statement delete "An in-depth
explanation...Training Guide."

02-01-1Q 2010 No Changes

~

REFERENCES:

STANDARD OPERATING PROCEDURE
4;
SUPPORT OPERATIONS
CRIMINAL LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

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SECTION: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

04-01-10 8 Page 1 of 7
SU8JEC71 EVENT:
EXCESS QUANTITY CASES

SCOPE
To provide guidelines for handling excess quantity controlled substance cases.
POLICY

An excess quantity case is defined as a controlled substance case for which a representative sample must be taken and reserved. The evidence will be photographed, analyzed, and handled in accordance with established laboratory procedures and Texas Drug Laws, and Safety Code section 481.160: Destruction of Excess Quantities. controlled substance cases will be analyzed by two analysts.

Note: If a latent print is requested, refer to the latent print section in

Evidence Handling
PROCEDURE

The receiving analyst and his/her co-worker should place the unique case identifier and initials on all exhibits.

- The analysts will ensure that the case is photographed. The photograph should reasonably demonstrate the entire case. If all containers cannot be encompassed in one photograph, overlapping photographs should be taken. If the case is processed in parts due to space or time constraints, then each part should be photographed and documented separately to represent the whole. Digital photographs are acceptable as long as individual items can be distinguished. Photographs should be labeled to include the unique case identifier and item designators, analysts' handwritten initials, and the date the photos were taken. A videotape may be taken at any time at the discretion of the analyst.

REFERENCES:

Texas Health and Safety Code Section 481.160: Destruction of Excess Quantities

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Weights of all items will be observed and verified by both analysts. All bundles will be grouped according to size and appearance. A reasonable packaging tare weight will be determined for each bundle grouping.

To determine a reasonable tare weight:

The packaging from at least one of the largest packages in each bundle group will be completely removed and weighed. At this point, the bundle should be broken apart to check for consistency throughout the whole bundle. The decision whether or not to open other bundles completely depends on apparent lightness, heaviness, or appearance will be at the discretion of the analyst.

•

If the total weight for the case is near or over the weight used as a cut-off in the Texas Drug Laws, the receiving analyst will determine appropriate weighing method.

•

The sampling and analysis of all exhibits will be served by both analysts. Refer to the CS-SOP 03 Analysis Section for the appropriate sampling and analysis procedures depending on the type of evidence submitted (powder, plant substance, liquid, etc).

After weighing and analysis of evidence is completed, the representative samples will be assembled and sealed. Both analysts will observe and verify the collection and weighing of representative sample and initial appropriately on the examination sheet.

To determine an appropriate representative sample:

1.

The representative sample will consist of a minimum of five separate containers randomly sampled from the total amount of evidence.

2.

If the contents of five total original containers meet the representative sample requirements outlined under Retention of Samples, these intact containers may be sealed as the representative sample. If less than five intact containers are available to provide the sample required, the analyst makes up the difference for the representative sample with samplings from the remaining excess quantity controlled substance. Refer to Retention of Samples for requirements to prepare representative samples for specific types of controlled substances.

3.

Evidence that consists of one single container of liquid will require the taking and preserving of only one representative sample.

REFERENCES:

Texas Health and Safety Code Section 481.160: Destruction of Excess Quantities

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4. Any items that are not bulk-wrapped (i.e., baggies, pipes, etc.) will be retained as part of the representative sample. An appropriate notation will be made on the worksheet under each item.

5.

Part of the representative sample should be composed of an intact parcel of the excess quantity case, if possible (i.e., one brick, one bundle, etc.).
If a large excess quantity case is composed of evidence from multiple addresses, retain a representative sample from each source.

At least one set of initials from all submitting office if available, and the receiving analyst or CER representative will be retain the representative sample. The initials will be either examples of the initials from the original packaging or a photograph of the initials. The representative sample should be labeled as "Representative Sample."

The remainder of the case will be packaged quantities as follows:
The container size for excess should be limited to forty pounds.

The following information should be on each container:
Analysts' initials
Case identifier;
Notations of "1 of 5," "2 of 5, etc." or the EMS item number (e.g., 02, CER1p4123, etc.) to identify multiple containers in the case; and
"Excess."

Information on the containers should be clearly visible and on two sides of the container. Use labels to place the

information on dark containers. All information on the plastic bags should be covered with tape. All bags should be deflated as much as possible.

RETENTION OF SAMPLES
Excess Quantity Plant Substance:

- Approximately 1.5 kilograms should be retained as a representative sample. At least five separate containers must be present (Health and Safety Code section 481.160).

- Fresh plant substance will be dried, and all roots, dirt, and stalks removed prior to weighing (stalks are the large woody stems that are negative for THC). At least

five separate containers must be saved.

REFERENCES:

Texas Health and Safety Code Section 481.760: Destruction of Excess Quantities

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EXCESS QUANTITY CASES CS-SOP 16 Page 4 of 7

- In the case of other excess quantity plant substance cases such as Knat, it may be necessary to retain the representative sample in the freezer.

Excess Quantity Powders:

- ~ One intact kilogram package and 4 small bags should be retained as a

representative sample. At least five separate containers must be present. If the excess quantity powder case does not contain kilogram packages, over 400 grams and at least 5 packages must be retained.

- For powder cocaine identified for federal prosecution, either kilogram packages should be retained as a representative sample.

Excess Quantity Liquids:

- At least 500 milliliters (at least 400 grams) should be retained as a representative sample (chemical precursors or liquid controlled substances).

- If the excess quantity liquid is in only one container, only one sample of at least 500 milliliters (at least 400 grams) should be retained.

Tablets and Capsules: ~

- At least 400 grams of an individual substance tablet or capsule should be retained as a representative sample. At least five separate containers must be present. For large non-controlled substance tablets or capsules, usually a small representative sampling is sufficient.

REPORTING

The report of an excess quantity case should follow the Reporting Guidelines Section as usual with the inclusion of the following footnote:

The Houston Police Department Crime Laboratory has photographed, determined the total weight of the substances and has retained representative samples as prescribed under the provisions of chapter 48.760 of the Texas Controlled Substance Act. The excess quantities will be destroyed 28 days after separate notification unless the Laboratory receives notice from the District Attorney's office before that date. The Houston Police Crime Laboratory will retain sufficient documentation as to the ultimate disposition of the narcotics.

REFER~NC~S:

Texas Health and Safety Code Section 481.~60: Destruction of Excess Quantities

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EXCESS QUANTITY CASES CS-SOP 16 Page 5 of 7
SUBMISSION TO CER

- The case folder (including photos) must be technically reviewed prior to its final submission to CER.

- The analyst will submit the entire case to the Centralized Evidence Receiving (CER) section for storage utilizing the following instructions:

1. The representative sample and the case folder will be personally delivered to CER personnel.

2. CER personnel will personally verify all portions of the case to be stored, both the representative sample and the excess quantities.

REFERENCES:

Texas Health and Safety Code Section 481.160: Destruction of Excess Quantities

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EXCESS QUANTITY CASES CS-S~P ~ 6 Page 6 0~ 7
MODIFICATION SUMMARY
DATE VERSION CHANGE

02-01-09 2009 New format for Headers and Footers
Section title changed from "Bulky Cases" to "Excess Quantity Cases" and throughout# section
Add References

p. 1 — Procedure:
Second bullet point expand section of photographs
and remove reference to guide.
p. 4— Tablets and Capsules
Remove second bullet "Any other unusual bulky cases should be brought to the attention of a supervisor."
p. 4-- Delete entries
orders for Excess quantities
p. 4— Submission
to CER:
First bullet point add "...technically reviewed prior to its
submission to CER."
e"4. CER will handle and file the case according
to R SOP"
e Checklist #for Bulky Cases

02-Q1-10
10 change references to "Lab number" to "unique case identifier"
throughout (per 09-30-09 memo).

p. 3— Add "Notations of "115, 215, etc." or "1 of 5, 2 of 5, etc."
or the EMS item number [001, 002, CER904123, etc.
to identify multiple containers of the same case"
p. 4— Tablets and Capsules add "At least five separate
containers must be present."
p. 4— Submission to CER:
Remove "2. A notation describing all the containers
including the representative sample should be placed on
the submission form above the chain of custody box CER
will use to receive the completed case (for example: rep
sample + two boxes + three bags)."

REFERENCES:

Texas Health and Safety Code Section 481.180: Destruction of Excess Quantities

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EXCESS QUANTITY CASES CS-SOP 16 Page 7 of 7

04-01-10 2010 p. 4— include footnote to be added to report for excess quantity

cases.

REFERENCES:

Texas Health and Safety Code Section 481.160: Destruction of Excess Quantities

~ _ ~ ' ~ STANDARD OPERATING PROCEDURESSUPPORT OPERATIONS
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CONTRQLED SUBSTANCES SECTION

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SECTION: DAT~ QF REVISIdN: REVISION NUMBER: PAGE NUMBER:
02-p1-9Q 6 Page 1 of2
SUBJECTIEVENT:
CLANDESTINE LABORATOREES ~
This Section is rescinded as of August 16, 2004. \

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NUMBER:

CLANDSTONE LABORATORIES CS-S-P 17 Page 2 of 2
MODIFICATION SUMMARY

DATE VERSION CHANGE

01-01-092009 New format for Headers and Footers.

02-01-022010 No Changes

REFERENCES:

STANDARD OPERATING PROCEDURES

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CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION
CATEGORY: DATE ISSUED: EFFECTIVE DATE: PROCEDURE NUMBER
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02-01-10 6 Page 1 of 3
SUBJECTIVE:~NT:
MONTHLY INVENTORY
SCOPE •/

~ed in the laboratory is
A monthly inventory of most of the controlled substances
~bers are used to track these
sent to the Department of Public Safety in Austin.
~hout the state. They are also
types and amounts of controlled substances seen

are#ory.
used to justify positions and expenditures for t c
PROCEDURE r~.
The monthly inventory sheet is to compile the data for controlled substances
received by each analyst. The amount of substances received each day is entered on

ed by each category. The following guidelines
the sheet by placing the amount
should be used:

. Those substances are listed by weights (such as cocaine, marijuana,

heroin, etc.) are listed using either grams, kilograms, or pounds; grams and
kilograms on the left, pounds on the right.

. All tranquilizers, synthetic narcotics, LSD, codeine tablets, and barbiturates are
listed by dosage unit (# of tablets or capsules) next to the appropriate category.

.
Methadone and codeine liquids are listed by volume in milliliters. If cocaine,
methamphetamine, or amphetamine are in liquid form, the amount is listed in
milliliters.

.
All designer drugs are grouped together and listed either by weight or dosage

unit.

REFERENCES:

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j MONTHLY INVENTORY CS-50P 18

•

PCP cigarettes are listed under # C1GS, and PCP liquids are listed by volume in millili#ers.

. The analyst's initials, the month, and #he year should be included at the top of the monthly in~entory sheet before t~rning it in to the appropriate person.

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MONTHLY INVENTORY CS-SOP 18 Page 3 of 3

MODIFICATION SUMMARY

DATE VERSION CHANGE

01-01-09 2009 New format for Headers and Footers.

p.1 - Remove bullet point 2 "Residues, cigarettes..."

p.1 - Remove bullet point 3 "For chemicals,..."

02-01-10 2010 No Changes

REFERENCES:

STANDARD OPERATING PROCEDURES

.. SUPPORT OPERATIONS
~~ CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

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Q4-01-10 8 Page 1 of 1 Q
SUBJECT EVENT:
REPORTING GUIDELINES

SCOPE

To establish standards for reporting the results from controlled substances, dangerous drugs, clandestine laboratory chemical substances examined by analysts at the HPD Crime Laboratory.

PROCEDURE ~

Reports are entered into the OLO based on the HPD incident number which is located at the top of the report. Additional information is entered in the appropriate fields: the EMS number or the laboratory number, and the analyst's (criminalist) name and title. After suspect enter the names of all suspects listed on the submission form. It is listed as "Inu Inu" (first name unknown, last name unknown), none, or unknown suspect as unknown. Under results of analysis at appropriate results will

REPORTING FOR ANALYTICAL RESULTS

HPD reporting guidelines for controlled substances are based on the laws and definitions provided in Chapters 481-485 of the Texas Health and Safety Code which contains the Texas Controlled Substances Act. The law determines the terminology used in reporting the identification of most controlled substances and requires the net weight of that substance to establish the penalty group.

Reporting Results of Controlled Substances and Dangerous Drugs

General Reporting Examples of Identification

1. Report the identification of a controlled substance as it appears in the Texas
REFERENCES:

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Controlled Substances Act.

2.

Precede the name of all substances identified with the word "Contains".
Marihuana and peyote will not be preceded with "contains" unless they contain other materials.

3.

If more than one controlled substance is identified in a sample, report them all after "Contains".

Examples: Contains Amphetamine and Methamphetamine
Contains Cocaine and Phencyclidine
Contains Cocaine and Marihuana

Identified in a sample,

4.

When a controlled substance and a dangerous drug are identified, the analyst should normally report only the controlled substance on the examination sheet. At the presence of the dangerous drug, at the discretion of the analyst, it may be necessary to report other substances identified for certain cases.

5.

If a sample contains only dangerous drugs, report all dangerous drugs identified. Report them using common generic drug name, not their pharmaceutical trade name, and include the notation that they are dangerous drugs.

Example for report: Contains Sildenafil — Dangerous Drug
Reporting Marijuana Seeds and Hashish
When a substance identified as marijuana as "Marijuana" (not

"cannabis") and report the weight in ounces or pounds.

2.

If a significant amount of an impurity, such as tobacco, is present in the marijuana sample (and cannot be readily separated), make a conservative visual or microscopic estimate of the percent of marijuana present, note this on the examination sheet, and report the total net weight in ounces or pounds. Report the substance beginning with the word "Contains" and add

an appropriate footnote:

Example: Contains Marihuana '

*Visually estimated to be 33~ of the reported weight

3. Report the results of the charred remains of marihuana {from pipes, s#ubs,
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ashtrays, etc.} as "Marihuana" and the weight as "trace" if microscopically identifiable marihuana is present. If insufficient physical characteristics are present to identify marihuana, then the results should be reported as "No controlled substance identified".

4.

For cases that consist of marihuana seeds only, they may be reported as "Marihuana seeds" and the weight in ounces. If no seeds germinate, report as "No Controlled Substance identified" with a footnote: "Marihuana seeds were identified and determined to be incapable of beginning germination".

5.

Report hashish and (liquid extracts as "Contains drocannabinol" and the weight in grams.

•

Reporting Peyote Samples

For plants visually identified as peyote and to confirm the presence of mescaline, report as "Peyote" with 'g grams. If the plant material cannot be visually identified as peyote or other samples, report as "Contains Mescaline" along with the weight in ~.

Reporting Mushroom Samples

Report psilocybin mushroom as "Contains Psilocin". Psilocybin may be reported if it has been identified by LC and FT~IR or TLC and a derivative procedure on the GC/MS.

Reporting Opioid Samples

Morphine, codeine and thebaine are the opium alkaloids that are controlled substances. Non-controlled alkaloids include papaverine, noscapine and narceine. Opium samples, including commercial preparations such as Paregoric, should be reported as "Contains Opioid" only if there is no heroin present and morphine and codeine are detected in combination with at least one of the other alkaloids. Samples which contain heroin should be reported as "Contains Heroin".

2. Alternatively, the results can be reported as "Contains Codeine, Morphine and (at least one other major alkaloid)" with a footnote stating: "These are commonly detected constituents of opium."

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Reporting Derivatives of Barbituric Acid

There are a number of derivatives of barbituric acid that are listed by name in the law. In those cases, report the name of the barbiturate identified (for example, "Contains Secobarbital"). If the barbiturate is not listed by name, such as butalbital, then it should be reported as "Contains a derivative of barbituric acid".

Reporting Weights and Volumes

-

If a controlled substance is identified in a powdered sample substance, or tar substance and the results are to be reported, report weight of the sample.

-

Report the weight of liquid samples if a concentration is identified. The volume may also be reported.

-

If the contents are identified and reported, include the weight of controlled substance tablets and capsules on test. If desired, the number of tablets and capsules may also be reported.

-

If a dangerous drug is identified sample (tablets, capsules, liquids, etc.), then no weight is necessary on test.

-

Except for marijuana, report net weight in grams if the sample ranges from 0.01 grams to 1,000 grams greater than or equal to 1,000 grams may be reported in kilograms. Weights less than 0.01 grams should be reported "Less than 0.01 grams". Residue amounts should be reported as trace.

- For marijuana samples weighing less than one pound, report the weight of marijuana in ounces. Report marijuana samples weighing more than one pound in pounds to at least one decimal place. If a marijuana sample weighs less than 0.01 ounces, the analyst should report the weight as "Less than 0.01 ounces".

Reporting Abuse Units

Report the number of abuse units of LSD samples as defined in HSC 481.002(50). Count and report the number of perforated blotter paper, tablets, gelatin wafers, sugar cubes, stamps or other single abuse units. If the blotter paper is not marked, each one quarter-inch square section of paper is considered a single abuse unit. If the sample is a liquid, 40 micrograms is one abuse unit.

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Miscellaneous

- Di~utants (diluent) and aduiterants should no# be reported on a routine basis. However, they may be reported a# #he discretion of the anaEyst, ~f requested by the submitting o~cial or prosecutor's office or if it is deemed necessary due to case circumstances.

The salt form of the dn.rg wifE not be repo~ted uNess that salt form has been properly identified using FTIR or other scien#ifca~ly ac~aep procedures. Likewise> the base form will not be reported unless the base fo een ~erified using FTIR or other scientifically accepted procedures. /~

- For certain substances, it is necessary to l

~ e orm present to establish

the appropriate penalty group or id~ ~ g. dextropropoxyphene, dextromethorphan, citalopram, and escita ;E3harmaceutical irrforma#ion ~s used to determine #he isomer farm pre the report should include an appropriate footnote, such as: ~~

"Isomer identified by information"

-

In tablets, capsu~es and liqui p ceutical preparations containing a cor~trolled substance, i# is sometE ssary to lcnw the amount of the controlled substance present to es bli e penaity group as stated in the Texas Controlled Substances Act. T~i present may be detennFned by accepted analytica~ quanti#ation pr~e es by a~ailable pharmaceutical information.

ff phamna 'cai ~ rmation is used (quantitation not performed), an appropriate footnote shou included in the report, such as:

"Pharmaceutical iden 'trf'rcation indicates not mor~ fhan 200 milligrams of c~adeine per 100 miNil'r~ers or ?00 grams and incl~des one or mor~s nonnaraotic actrve medrdnal ingredients." or

"Pharmaceu#ica! rdenification indicates 800 milligrams per dosage unit."

When- pharmaceutical information is not available (as in the case of a crushed tablet or codeine 1 promethazine. cough syrup poured into a soda) and quantitatfon is not performed, then report the substanoes ident~ed in the exhibit after °Contains"

Example: Confains codeine and promethazine

REFER~NCES:

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Contains dihydrocodeinone and acetaminophen

- Steroids and steroid esters should be reported by the steroid alcohol name.

Example: Contains Testosterone or Contains Nandrolone

+

If a sample is examined for the presence of an abusable volatile chemical as listed in HSC 485, and one is identified, then report the results of the substance identified with the notation that it is an abusable volatile chemical. No weight is necessary on the report. ~

Example: Contains Toluene — An Abusable Volatile Chemical

•

Items for which visual examination by two notes that no sample / residue is present for analysis should be reported as "no analysis performed (no visible sample),"

When field detectors are received with or evidence to analyze, they should be reported as "No unprocessed material for analysis."

•

Exhibits that are not analyzed as "Retained with no analysis" and no weight needs to be reported. Appropriate footnotes may be added at the analyst's discretion, such as:

"All remaining submitted with this case will be retained without analysis. If analysis is needed for prosecution purposes, contact this laboratory."

- Samples may be reported as "No controlled substance identified" after the sample has been subjected to sufficient analytical examinations. No weight needs to be reported and an appropriate footnote may be added at the discretion of the analyst.

If a substance has been subjected to preliminary pharmaceutical identification without analytical confirmation, the report will reflect "Indication [substance]".

If a dangerous drug or over the counter substance is indicated, then the report will include the notation that the substance is a dangerous drug or an over the counter product. The notation "Pharmaceutical identification only" may be added as in the following example:

Example: Indication Amitriptyline — Dangerous drug "

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Indication Acetaminophen — Over the counter *

* Pharmaceutical identification only

In the situation where a confirmation test is unavailable by the laboratory to support pharmaceutical identifications (insulin, human growth hormone, new products without published characteristics), the report should include the available information with an appropriate footnote:

Example: Indication Levothyroxine — Dangerous *

* Pharmaceutical identification complete analysis is not possible by this laboratory.

For cases processed according to the Entity Cases Section where photographs and representative samples retained, the following footnote will be added to the report: ~_

The Houston Police Department Laboratory has photographed, determined the total weight of the substances and has retained representative samples described under the provisions of chapter

481.160 of the Texas Controlled Substance Act. The excess quantities will be destroyed 2 days after separate notification unless the Laboratory receives notice from the District Attorney's office before that date. The Houston Laboratory will retain sufficient documentation as to the disposition of the narcotic(s).

• Reporting guidelines for Disposed, Dismissed, and Destroy cases can be found in the Disposed, Dismissed, and Destroy Case Guidelines Section.

• Other footnotes may be added to the report at the discretion of the analyst when circumstances mandate it.

Footnotes

The following is a list of the footnotes that will be available with a shorthand abbreviation typed into QLQ after "--F" (SOE character followed by F space then the identifier).

• Pharmaceutical identification only. No chemical analysis performed. If analysis is

required for prosecution purposes, please contact this laboratory at least one week

REFERENCES:

SUBJECT/VENT: PROCEDURE PAGE NUMBER:

NUMBER:

REPORTING GUIDELINES CS-50P 19 Page ~ of 10
prior to trial date ~- FPHI y.

- No more than 9.5 grams of codeine, or any of its salts, per 700 milliliters or not more than 94 milligrams per dosage unit, with one or more active, nonnarcotic ingredients in recognized therapeutic amounts (- F CQT).

- Not more than 300 milligrams of dihydrocodeine, or any of its salts, per 900 milliliters or not more than 15 milligrams per dosage unit, with one or more active, nonnarcotic ingredients in recognized therapeutic amounts (- F DHY).

-

Not more than 200 milligrams of codeine per 100 milliliters or 200 grams and includes one or more nonnarcotic active ingredients in - F CQL.

-

All remaining evidence submitted with this retained without further analysis. If analysis is needed for prosecution, please contact this laboratory (- F RET).

- The HPD Crime Laboratory is + b CLD / LAB and the Texas DPS.

Laboratory policy requires that analytical results undergo a technical review by a second qualified analyst.

Other Suggested Footnotes (DPS)

- A Dangerous Drug)

- The during analysis.

- Weight includes the weight of the rolling paper.

- No analysis was performed.

- Substances commonly found in opium.

-

An analogue of gamma-Hydroxybutyric Acid (gamma-Hydroxybutyrate),

- Specialized footnotes may be used with the approval of a supervisor.

REFERENCES:

-

SUBJECT/EVENT: PROCEDURE PAGE NUMBER:

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REPORTING GUIDELINES CS-SOP 19 Page 9 of 14

M~D~FICAT1pN SUMMARY

DATE VERSION CHANGE

02-01-09 2009 New format for Headers and Footers

Add References

p. 1 -- Scope

Delete "Detailed instructions for accessing the 0~.0 system are provided in the ining Guide."

p. 2— General Reporting Examples Identification

2. Remove refere t r g quantitated controlled substance resul

3. Remove refe t eporting quantitated controlled substance re ts. o~e Example "Cocaine (10°~) and Meth ne {10°~)". Add Example "Cantains Cocain n arshuana° ~

p. 3— Re ~

yote Samples

°... Contains Mescaline, d ,
lo rnnt the weight in grams."

4— ove conversion fac#ors for English to metric weights
nd put in Training Guide

p_ — Third buflet point change °... and no weight ~s needs to
be repor~ed... °

Sixth bullet point remove examples of destroy cases
and pled out cases for reporting °Indication [substance]"

p. 7— Add bullet point referencing Disposed, Dismissed, and
Destroy Case Guidelines Section I

REFERENCES:

SUBJECT/EVENT: PROCEDURE PAGE NUMBER: ~

NUMBER:

REPORTIN~ GUIDEUNES CS-SOP ~9 Page 10 of ~0
DATE VER510N CHANGE

02-01-10 20'~ 0 Change °report#ed as Retained' to

"reported as Retained with no analysis" throughout.
Change "reported as No con#rolled substanceA to
"reported as No controlled substance identi#iedA throughout
p_ 1 -- Procsdure

Add "Reports are entered i the 0[.O sys#em based
on the HPD incident num is located at the top
af the report. The foll dd nal i~ormation is
entered in the app opr : the EMS assigned
item number or number, and the analyst's
(criminalist) name ' e."

p. 3-- Reporting O um a ples ~1)
Add " s contain heroin should be reported
as "Co ' s roinp.

p. 4— R
rti eights and Volumes modify °Weights
than or equal to 1,000 grams should be reported
s rams.° to "Weights greater than or equaf #o
grams may be repo~ted in grams or fn kilograms."
er 12-28-09 memo)

— Th~rd bullet point add as an example uPharmaceutical
identification indicates 800 milligrams per dosage unit.A

p. 6— Fourth bulle# point delete as art example
°Pharmaceutical ir~ormation indicates that no controlled
substance is present.°

Sixth bullet point reword for reporting preliminary
harmaceutical identification results.

Q4-01-1(} 20'~ 0 p. 3(4} — Change "No controlled substanceA to °No controlled
substance ident~edA tper 02=11-10 memo)

p. 6— Si~h bullet point clari#ied so that the notation
"Pharmaceutical identification onl~' is an optiona!
addition on the report. (per 02-11-10 memo)

p. 7— Include bul3et point for footnote to be added to report for
excess uanti cases.

RE~ERENCES:

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~ M ~

STANDARD OPERATING PR4CEDURES

SUPPORT OPERATI~NS

CRIME EABORATORY DIVISION
CONTR~LLED SUBSTANCES S~C~'ION
EFFECTIVE DA7E: PROCE~L1ftE NIJM6ER
CAT~GORY: DA~E I\$SU~U:
a2-01-10 CS-SOP 20
01-01-06
PAGE NUMBER:
SECTION: aATE OF REVISION: REVISIO~I NUMBER:
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02-01-10
SUBJECTIEVENT:
ABBREVIATIONS

~
SCOPE
To provic~e a list of useful abbreviations.
ABBREVIATIONS

~

~~'Balance

I~ase extraction
AB.....: ~
AlBextr

ra-Chem Library Search
ACLS.....~~

.~~mphetamine
amph
approxAp~aroximately

..Administrative re~iew
AR
...Abusable Valatile Chemical
AVC
...Bulky Balar~ce
BB.....

.....1,4-butanediol
1,4-BD

..... Benzylpiperazine
BZP

~ -

c..... Containing
cig..... Cigarette
cigstubCigarette Stub
coc.....Cocaine
CPPChloro-phenylpiperazine
DBZP..... 1,4-DiEaenzyfpiperazine
DD.....Dangerous Dr~g
DIBDrug Identification Bible
DMS..... Dimethylsulfone

D.O Des#roy
EDIA Evidence Destroyed in Ana~ysis
EMSEvidertce Management System
REFERENCES:

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NUMBER:

ABBREVIATIONS CS-S-P 20 Page 2 of 4

est..... Estimate(d)
Extr..... Extracted or Extraction
FTIR..... Fourier Transform Infrared {Spectrophotometry}

g..... Grams
GBL..... gamma-butyrolactone
GC..... Gas chromatograph
GHB..... gamma-hydroxybutyric acid{gamma-hydroxybutyrate}
Ind..... indication
JIMS..... Justice Information Management System
kg..... Kilograms
lb..... Pounds
L..... liters
iIMS..... Laboratory Information Management System
LSD..... Lysergic Acid
Mari..... Marijuana
Marih.....
MDA..... amphetamine

metfi-amphetamine
MDMA..... N-ethylamphetamine
MDE.....
MDP2POL..... phenyl-2-propanol
meth.....
ml.....
MS..... mass spectrometer
NAM..... No acceptable match or Not an acceptable match
NAP..... No Analysis Performed
NCS..... No Controlled Substance

.. '..... Negative
OO..... HPD On-line Offense Report Access System
oz..... Ounces
PCP..... Pencyclidine
PDR..... Physician's Desk Reference
PHI..... Pharmaceutical Identification
pkg..... Pac-Cage

Positive
pos.....

Prometh..... Promethazine
 PS Plant Substance
 ~ Retention fiactor (TLC)
 RT..... Reten#ion time
 STD..... Startdard
 TB Top-Loading Balance
 TFMPP..... 1-(3-Trifluoromethylphenyl)piperazine
 R~F~R~NCES:

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ABBREVIATIONS C5-SOP 20 Page 3 of 4

THCTetrahydrocannabinol

TLCThin layer chromatography

TRTechnical review

UVNISUltraviolet-NISible (Spectrophotometry}

wt..... weight

REFERENCE5:

SUBJECT/EVENT: PROCEDURE PAG~ NUMBER:

NUMBER:

ABBR~VIATIONS CS-50P 2Q Page 4 nf ~I

MODIF~CATION SUMMARY

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STANDARD OPERATING PROCEDURES

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• ~~~~€ -. SUPPORT OPERATIONS

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CRIME LABORATORY DIVISION
CONTROLLED SUBSTANCES SECTION

CATEGORY: DATE ISSUED: EFFECTIVE DATE PROCEDURE NUMBER

1-21-08 02-01-1 Q CS-SOP 21

SECTION: DATE OF REVISION: REVISION NUMBER: PAGE NUMBER:

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suat-crr-vENr:

CONTROLLED SUBSTANCES SECTION WORKSHEET (WEEKLY)

SCOPE ~'

To provide guidelines for the counting of items the Houston Police Laboratory Controlled Substance Section Worksheet is referred to as the Weekly Worksheet. The information from the worksheet will be entered into the case management system to document the filing of cases and to assist with monitoring section productivity.

CONTROLLED SUBSTANCE SECTION

•

The analyst's name is entered below.

•

The date starts on Monday and goes through Sunday of each week except at the beginning or end of . Do not put parts of two months on one sheet. As a result, one sheet will have only one or two days on it.

•

The unique identifier is placed in the column that has Lab Number at the top. If the case being reported has been reported previously or belongs to another analyst (usual situation for analysts in training), put parentheses around the number.

ITEM COUNTING GUIDELINES

• In the column headed # Items record the number of items analyzed. Only items tested are counted. For example, if 100 ziplocks of powder are received, but only 10 are tested, then the number of items to be counted is 10. If 2000 tablets are listed but only 50 are spot tested, then the number of items is 50. If individual chunks are sampled for separate spot testing from a single container of chunk substance, then the number of items is still 1.

REFERENCE:

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WEEhLY SWEET --CS-SOP 21 Page 2 of a

- If pharmaceutical identification is performed for a case without spot tests, then the number of items is based on the number of containers and the different tablet/capsule logos. For example, pharmaceutical identification is performed on 5 bottles of tablets each having the same tablet logo; therefore, the number of items to be counted is 5. In another case one bottle contains tablets with three different logos; therefore, the number of items to be counted is 3.

On visual negatives count items that routinely have a residue such as spoons, syringes, matchboxes, etc. Do not count rolling papers, bags, coins, pieces of paper, etc.

TEST COUNTING GUIDELINES

-

In the column headed # TESTS record the total number performed in the analysis of the case. Add the number of samples to the number of microscopic exams to the number of instrumental exams.

HOW TO COUNT TESTS:

Marthuania - Add the number of microscopic exams to the number of Duquenois spot tests and the number of Duquenois spot tests performed.

Spot Tests - One test for each test performed. For example, if 240Q tablets are received and 50 Ferri and 50 Marquis spot tests are performed then the number of items is the number of tests performed is 100.

UV — One substance identified by UV. UV with a shift counts as one test. as two substances and a UV is obtained for each substance counts as two tests (ex. codeine and acetaminophen). No acceptable a negative result counts as one test.

FTIR - One test for each substance identified by IR. No acceptable match counts as one test.

GCIMS - One test for each sample run on each instrument.

TLC - One test for each solvent system used and one test for each standard spotted.

Microcrystalline - One test for each type of microcrystal from each item.

PHI - Any pharmaceutical ID on an item (tablet, capsule, ampoule, etc.) from an accepted source (PDR, Logo Search, Mexican PDR, etc.) is counted as a test.

Just writing down the markings from the item is not a test. An unsuccessful

REFERENCES:

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4 WEEKLY SHEET CS-SOP 21 Page 3 of 4

attempt at identification counts as one test and is documented on the examination sheet. Pharmaceutical identification will be counted as one test per column on the examination sheet no matter how many dosages exist. For example, if 500 tablets are listed in one column, only one pharmaceutical test may be counted. Visual - Visual negative is counted as one test for each item with a visual negative.

IDENTIFICATION GUIDELINES

Enter the identification of the item(s) in the case. Use the headings at the top of the columns as a guide for the information to be included.

- Some items may only require a check mark to indicate identified (e.g. marijuana, cocaine, or PCP).
 - Tablets will require the number present as well as whether the tablets were indicated by pharmaceutical identification or confirmed by complete analysis.
 - Substances which are not specified are to be noted in the column labeled OTHER. This includes not in N " or "No Controlled Substance".
- #### MISCELLANEOUS COUNTING GUIDELINES

- When items are received, counting the number of items is based on the documentation on the examination sheet. One item is counted for each column of items. The number of tests will be zero. If retained items are analyzed at a later date, the number of items and the number of tests will be counted.
 - If an item is reported as "No Analysis Performed", then it will be counted as one item, but with zero tests.
 - When evidence is re-examined by another analyst the items are re-examinations performed on the exhibits are counted on the weekly worksheet as for other cases.
 - If you have any questions about how to count the number of items, tests, or note identifications, then consult with a supervisor.
- #### REFERENCES:

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NUMBER:
WEEKLY SHEET C5-SQP 21 Page 4 of 4
MODIFICAT~ON SUMMARY

DATE VERSION CHANGE
0"~-01-092009 New format for Headers and Footers.
02-01-10 2010 p. 1-- Controlled Substance Section Worksheet

Third bullet point change "lab number o# the case" to

"unique case identifier". (per 09-30-09 memo)
REFER~NCES:

STANDARD OPERATING PROCEDURES

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.. SUPP~riT OPERATIC~~iS

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CRIME LABORATORY DIVISiON

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CONTROLLED SUBSTANC~S SECTION

CATEGORY: DATE ISSUED: EFFECTIVE DA~: PROCEDURE NUMBER

01-21-08 02-01-10 CS-SOP 22

S~CTION: DATE OF REVISION: ~ REVISION NUMBER: PAG~ NUMBER:

o2-a~~o l z Page 1 of 3

SUBJECT/~VENT:

RE-ANALYSIS OF CASES

SCOPE

To pro~ide guidelines for conducting fe-analysis o# ca s~Q 4Zl~iarious circumstances.

~

RE-ANALYSIS FOR PURPOSES OF TESTIFYI ~N O RT

e -analysis of cases when the origi~ial

The following guideline is provided to aid i

analyst is not a~ailable to testify in court.

. The Controlled Substance anager or designee will assign t~e case to

an ana~yst who wil~ recei~ ev ence from Centralized E~idence Recei~ing.

. The new analyst wil e case following normal procedures for ana~ysis

and use a ne x in n sheet for documen#ation.

•

Report ~n s iR upplemental rsport using the following wording:

"On (date), Title (name), PR# was requested to re-anaiyze evidence in this offense for rhe pu~pose of testifying in an upcoming trial. "

RE-ANALYSIS FOR ON-G~ING REVIEW OR INVESTIGATtON

The following guide~ine is provided to aid in the re-examination and re-anafysis of cases re~iewed as a result of an on-going re~iew anc!!or in~estigation.

. Recei~e evidence from Contro~led Substance Section Manager or CentraHzed

Evidence Recei~ing section staff, as appropriate.

. Photograph e~idence en~elope wit~ seals

REFERENC~S:

CRIME LABORA70RY S4P, CORRECTIVE ACTION SECTkON

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RE-ANALYSIS OF CASES CS-SOP 22 Page 2 of 3

-

Photograph contents

- Inventory and weigh items.

The analyst will document discrepancies with the Quality Assurance Manager if differences are detected in weights, counts, or presence of discreet items. This documentation will take the form of an interoffice memo, as described in the Laboratory Standard Operating Procedures, Corrective Action Section.

-

Proceed with re-analysis of items, as determined by original examination sheets. Duplicate the work conducted previously.

-

Report findings in a supplemental report using the following:

"On (date), Title (name), PR# re-analyzed this as part of an on-going quality assurance review."

REFERENCES: _____

CRIME LABORATORY SOP, CORRECTIVE ACTION SC-710N

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RE-ANALYSIS OF CASES C5-S-P 22 Page 3 of 3
MODIFICATION SUMMARY
DATE VERSION CHANGE

01-01-09 2009 New format for Headers and Footers.

Add References

p. 2— Second Bullet point modify "This document will
take the form of an interoffice memo, as described in the
Laboratory _ . _ , _ _ ;
02-0--o | zo-o No Changes

~

REFERENCES:

CRIME LABORATORY SOP, CORRECTIVE ACTION SECTION

STANDARD OPERATING PROCEDURES

1. PURPOSE: SUPPORT THE PERIODIC
CRIME LABORATORY DIVISION

R
CONTROLLED SUBSTANCES SECTION

CATEGORY: ~ DATE 15SUED: ~ EFFECTIVE DATE: ~ PROCEDURE NUMBER

01-01-09 ~ 02-01-10 ~ CS-SOP 23

SECTION: ~ DATE OF REVISION: ~ REVISION NUMBER: | PAGE NUMBER:

02-01-10 Page 1 of 5

SUBJECTIVEEVT:

DISPOSED, DISMISSED, AND DESTROY CASE GUIDELINES

SCOPE ~f

To provide guidelines for identifying case status, as processing the evidence in cases which are identified as Disposed (DISP), Dismissed, Destroy, or for which a clear status is not available.

IDENTIFYING CASE STATUS

1. Case status can usually be identified through JIMS and OLO as Act+ve 1 DISP 1

DISM or a combination of the e.

-

Active cases are those which have pending court dates for one or more defendants. Information obtained from JIMS 1 OLO referencing this status should be included in case file and labeled with the unique case identifier and the initials of personnel researching the status.

-

For D 1 D M cases information obtained from JIMS / OLO should be included in case file to reference the following: HPD Incident #, Cause #, Defendant, Charges, and Status. Printouts from JIMS / OLO should be labeled with the unique case identifier and the initials of personnel researching the status.

.

The status of a case may not be readily available by reviewing JIMS or OLO or it may be determined that no drug charges have been filed for a particular case. Examples include Juvenile, Investigative Narcotics, Homicide, or cases filed outside of Harris County. Available information regarding the case status should still be included in the case file and labeled with the unique case identifier and the initials of personnel researching the status.

REFERENCES: ~

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DISPOSED, DISMISSED, AND DESTROY CS-SOP 23 Page 2 of 5
CASE GUIDELINES

PROCESSING OF ACTIVE CASES

- If a case is found to be active for all listed defendants, then it should be processed according to the Analysis Guidelines Section and the Reporting Guidelines Section.

- If a case has multiple defendants with a combination of status (Active / DISP 1 DISM), then the Active evidence should be processed and the remaining evidence may be retained. The JIMS status printouts should be included in the case file for the DISP / DISM evidence to support the decision to retain those items.

PROCESSING OF DISPOSED CASES

- If a case is identified as Disposed or Dismissed or defendants, then only the items associated with the Disposed evidence be processed. Items associated with the Dismissed evidence may be held.
For items associated with charges that be disposed, at least one positive test is required to report that a substance is indicated. At least two positive tests, one of which must be either FTIR or , are required to report that a substance is identified. For plant, positive microscopic and chemical screening is sufficient for the identification of marijuana.

- A weight should be recorded items analyzed with the exception of items which are or which contain residue (these are to be noted as "trace").

- For items analyzed the report should contain a description of the evidence, the weight, and the type of analysis. If the analysis performed indicates the presence of controlled substance, the report will reflect "Indication [substance]". Sufficient analysis has been performed to identify the presence of a controlled substance, then the report will reflect "Contains [substance]" or in the case of marijuana the report will reflect simply "Marijuana". If a dangerous drug is indicated or identified, then the report will include the notation that the substance is a dangerous drug. For items where a substance is not indicated or identified as a controlled substance or dangerous drug, the report will reflect "No controlled substance identified".

- The report should also include the associated Cause # and Status for each listed

de#endant.

- . A technical re~iew (TR} should be performed as for Active cases.
- . An accreditation footnote should be included in the report as usuat.

REF~RENCES:

SUBJECT EVENT: PR~C~DUR~ PAGE NUMBER:

NUMBER: '

DfSPOS~D,_DISM155ED, AND DESTROY C~-SQ° 23 ~a~ya 3 ~ 5

' CASE GUIDELINES

PROCESSING ~F DISMISSED CASES

- For cases where all of the evidence has been identified as Dismissed for all defendants, No analysis is necessary. A report should be generated in OLO noting that no analysis has been performed due to the dismissal of charges per JIMS.

-

The report should also include the associated Cause # for each listed defendant.

-

The individual entering a DfSM case report will perform an administrative review

~

(AR). There should be a secondary AR to verify the information in the report is correct and that the printouts from JIMSIOLO are included in the case file. No technical review (TR) is necessary since no analysis is performed.

-

No accreditation footnote is needed for DIS ~ since no analysis is performed.

PROCESSING OF CASES WITHOUT A CLE/TA S . ~!

-

After cases for which the status is undistributable (by reviewing JIMS or OLO or fax which no drug charges have been passed 180 days from initial receipt into the Laboratory, they must be analyzed following the same analytical and reporting procedures as for these Cases (see above).

-

A weight should be recorded for all items analyzed with the exception of items which are or which contain these are to be noted as "trace").

-

If an associated Cause # is available for listed defendants, it may be included in the report. For example Homicide or DWI cases may have a Cause # for the charged offense but no drug charges.

-

A technical review (TR) should be performed as for Active cases.

-

An accreditation footnote should be included in the report as usual.

- if additional information regarding the case status becomes available at a later date or if a request for a complete analysis is received, the case is to be handled per the instructions of the Section Manager or designee.

PROCESSING OF DESTROY CASES

-

Some cases are received and tagged as "DESTROY" by the submitting officer.
These are usually cases where there are no charges being filed and the offense is
REFERENCE:

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CASE GUIDELINES

listed as "found property", "investigation narcotics", etc. There may be a notation such as "ADA Smith refused charges".

- Most of these cases will be filed in CER with no analysis. In the event that a destroy case is distributed for analysis (usually "investigation narcotics" charges) the same analytical and reporting procedures should be followed as for Processing of Disposed Cases.

-

A weight should be recorded for all items analyzed with the exception of items which are or which contain a residue (these are to be noted as "t e"). If the gross weight of an item including packaging is used, this should be on the examination sheet and on the report.

. A technical review (TR) should be performed for cases.

. An accreditation footnote should be included as usual.

Any questions regarding procedures for analysis, or reporting of results for Disposed, Dismissed, Destroy, or uncertain cases should be directed to the Section Manager or designee.

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REFERENCES:

SUBJECT/EVENT:
DISPOSED, DISMISSED, AND DESTROY
CASE GUIDELINES

MODIFICATION SUMMARY
DATE VERSION

01-01-09 2009

02-d1-1Q 2010

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REFERENCES:

HOUSTON POLICE DEPARTMENT
CRIME LABORATORY

DOCUMENT AUTHORIZATION

Requestor: James T. Miller Date: Jun~ 10 2009

X Existing Version: CS-TG Version 2009 Modules 09 — 22

^ New If new document, briefly describe document contents.

This is the complete version of the 2009 Training Guide revision.
Submit hardcopy or electronic documentation to the Quality Assurance Manager
Signature: , ate G ~ ~ ~ w

Review

Q~i ppro~e / ^Reject

Date: (~ - I C, - ~ S

^Approve / ^Reject

DApprove / ^Reject

Comments:

~

Manager Review

DApprove / ~Reject~ : Date: ~ 7Q~ j

Plan for implementation:

Comments:

Laboratory Director

^Approve I ^Reject N C~ Date:

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HOUSTON POLICE DEPARTMENT CRIME LABORATORY
Controlled Substances Training Guide CS-TG
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Microcrystalline Tests MODULE 06

Midterm UVritten Exam •, • MODULE 07

Measurements and Sampling

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En~cti~e aat~ o2-os-as

HOUSTON POLICE DEPARTMENT CRIMINAL LABORATORY
Controlled Substances Training Guide MODULE 01
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CONTROLLED SUBSTANCES TRAINING SYLLABUS AND CHECKLIST
Trainee Employee Number
Trainer Date Training Begins

Date of Trainer 2
Topic Receipt or Trainee
Completion Initials

Module 01 - Introduction and General Orientation N/A N/A
Tour of Crime Lab Facilities and Assigned Work Area
Receive Introductory Materials including:

Lab Quality Assurance and SOP (QASOP)
and Relevant HPD General Orders (GO)
Controlled Substances Training Guide (TG) and SOP
Review of Training Guidelines
Review of Training Syllabus and Checklist
Review of Skills and Knowledge Questionnaire, etc.

Review Departmental and Lab Functions
HPD Organization Chart
Crime Lab Organization Chart and Location:
GO 10Q-06 — NPD Mission Statement
Lab QASOP — Crime Laboratory

SOP 01 — Section Mission Statement and Objectives
Safety Training:
Receive Safety Manual
View Safety Videos
Receive Safety Equipment

Accreditation:
Discuss accreditation process for Texas Labs
Receive and review current ASCLD-LAB manual

Review Lab QASOP and relevant HPD GO's
Effective date 02-09-09

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Controlled Substances Training Guide MODULE 01
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Miscellaneous:
Parking Permit
ID Badge and Employee Number
Computer Access and Email
Photo for Lab Personnel Record
Statement of Qualifications
Receive Security Keys and Access Codes
Attend New Employee Mandatory Training

Observe general casework analysis {on-going} N/A N/A

Module 02 - History and Control of Drugs of Abuse N/A N/A
Independent Reading
Observe HPD Cadet Narcotics Training and Review
Federal Controlled Substances Act and Texas
Review TG Module 02 Material
Review history of Federal Drug Laws and CS
Receive and review current Texas Health Agency orders including

Chapters 481 - 486 and Controlled Substances
Controlled Substances Training N/A N/A

Module 03 - Chemical Control of Drugs
Independent Reading
Review TG Module 03 - Chemical Control of Drugs and Structures
Receive TG Memoranda of commonly encountered Substances

Module 04 - Pharmaceutical Identification of Drugs
Independent Reading
Review following sections:
TG Module 04 Material
Complete Pharmaceutical ID Practice Worksheet
SOP 03 — Analysis Guidelines for Tablets and Capsules
SOP 20 — Abbreviations
SOP 05 — Examination Sheet
Review Examination Sheet Documentation for Pharm ID

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Controlled Substances Training Guide MODULE 01
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-Date of Trainer &
Topics Receipt or Trainee
Completion Initials

Module 05 — Chemical Screening /Spot Tests
Independent Reading
Review following sections:
TG Module 05 Material
SOP 03 — Chemical Screening Tests
SOP 12 -- Reagent Quality Assurance
SOP 01 — Standards and References

Prepare Reagent Bottles for Work Area
Complete and Review Practice Samples for Spot Tests
Review Examination Sheet Documentation for Spot Tests

Module 06 — Microcrystalline Tests
Independent Reading
Review following sections:

TG Module 06 Material including In-house Handout
SOP 14 — Microcrystalline Tests

SOP 12 — Reagent Quality Assurance
Complete and Review Practice Samples for Microcrystalline Tests
Review Examination Sheet Documentation for Microcrystalline Tests

Module 07 -- Midterm Written Exam
Receive Study Guide and Review with instructor
Take Written Midterm Examination and P

Module 08 — Measurement Sampling

Independent Reading
Review TG Module 08 Material
SOP 06 — Instrumentation, Maintenance {Balances}
SOP 03 — Analysis G (Sampling of Evidence)
Complete and Review Practice Exercises
Review Examination Sheet Documentation for Weights

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H~U~TON POLICE DEPARTMENT CRIME EAB4RATQRY

Controlled Substances Training Guide MODULE 01
Sub'ect: Introduction and General Orientation Version 2009

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Date of Trainer ~
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Com letion Initials

Moduie 09 — UV / VIS Spectrophotometry
Independent Reading
Review following sections:

TG Modufe 09.i Material (Qualitative only)

SOP f 0— UVNIS Spectrvphotometry (Qualitative oniy)
SOP Ofi — Instrt~ment Performartce and Mair~tenance {UV}
TG Module Og.2 -- UVNIS Shimadzu

Complete and Re~iew Practice Samples for Qualitative UVNIS

Review following section:
TG Module 09.1 Material (p~antitati~e only}
SOP 10 — UVNIS Spectrophotometry (Quantitation o~ly)

Comple#e and Review Practice Samples for Quantitati~e UVNIS
Receive Stud Guide for UVNIS and Review with Trainer

Module 10 — Separations and Extractions
Independent Reading
Review TG Module 10 Material
Prepare solvenis and su~lies for extractions in ~
Compfete and Review Prac#ice Samples for Extr i
Disauss and Practice Conway Extraction
Review Examination Sheet pocurrEer~tatio , r NI and Extractions

Receive Study Guide for Extrac#ians an Rev fth Trainer
Module 1'i — IR / FTIR Spect ph try
Independent ReadEng and Tr
Review following secti

TG Moduls 11.1 Materi
SOP 09 — FTIR trame
SOP 08 — Instrumen ance and Maintenance (FTIR)
TG Moduie ~ 1.2 — FTIR Thermo

Complete and Review Practice Samples for F71R
Review Examination Sheet Documentation for FTIR
Receive Study Guide #or ~TIR and Review with Trafner

Module ~2 — Thin Layer Chromatography
Indepe~dent Reading

Review following sections:
~'G Module 12 Material
SOP 15 — Th~n Layer Chromatography

Compfete Practice Sarnples for TL.0

Review Examination Shset Doceamentation for TLC
EfFective~date 02-09-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY
Controlled Substances Training Guide MODULE 01
Subject: Introduction and General Orientation Version 2009

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Date of Trainer &
Topics Received by Trainee
Completion Initials
Module 13 - Gas Chromatography / Mass Spectrometry
Independent Reading

Review following sections:
TG Module 13.1 Material
SOP 05 - Gas Chromatography/Mass Spectrometry
SOP 06 - Instrument Performance and Maintenance (GC/MS)
TG Module 13.2 - GC/MS Agilent
TG Module 13.3 - GC/MS Shimadzu
TG Module 13.4 -- GC/MS Varian

Complete and Review Practice Samples for GC/MS
Review Examination Sheet Documentation for GC/MS
Receive Study Guide for GC/MS and Review with Trainer

Module 14 - Anabolic Steroids
Independent Reading
Review following sections:

TG Module 14 Material
Complete and Review Practice Samples for Anabolic Steroids
{include discussion of unique circumstances for analysis procedures}

Module 15 - Marijuana and THC
Independent Reading
Review of Controlled Substances Marijuana and THC
Review following sections:
TG Module 15 Material
SOP 13 - Chemical Tests (Duquenois / Duquenois-Levine)
SOP 12 - Reagent Qualification
SOP 03 - Analytical Methods (Plant Substance)

Complete Practice Samples for Plant Substance and Hashish (if available)
Review Examination Sheet Documentation for Marijuana and THC

incl~din Marihuana Checkll5t
Effective date 02-09-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY
Controlled Substances Training Guide MODULE Q7
Subject: Introduction and General Orientation Version 2009

Page 1 of 14
Date of Trainer &
Topics Received or Trainee
Completion Initials
Controlled Substance Analysis of Evidence N/A N/A
Module 6 — Evidence Handling

Review following sections:
Lab QASOP — Evidence
SQP 02 — Evidence Handling
TG Module 16 Material

Review Submission form documentation
Review Latent Print forms documentation
Review Property Room forms documentation

Observation of Centralized Evidence Receiving Operations
Module 7 -- Analysis Guidelines

Review following sections:
TG Module 17 Material
SOP 03 -- Analysis Guidelines (a!!)
SOP 22 — Re-Analysis of Cases
SOP 23 — Disposed, Destroyed, and Destroyed
SOP 05 — Examination Sheet (aU)

Module 8 — Reporting of Results

Review following sections:
TG Module 18 Material
SOP 19 — Reporting Guidelines

Module 9 — Case File Division

Review following section
TG Module 19 Material
Lab QASOP -- Case File
SOP 04 — Case Documentation
SOP 21 — Weekly Sheet

50F~ 18 — Monthly Sheet

Module 20 — Monitored Analysis Performed by Trainee
Trainer observed analysis of at least 14 plant substance cases
including receipt of evidence, analysis, reporting of results,
weekly sheet and monthly sheet documentation, and return of evidence.
Trainee observed analysis of at least 10 controlled substance cases
including receipt of evidence, analysis, reporting of results,
weekly sheet and monthly sheet documentation, and return of evidence.

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HOUSTON POLICE DEPARTMENT CRIME LABORATORY

Controlled Substances Training Guide MODULE p1

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Date of Trainer Signature

Topics Received by Trainee

Comments/Initials

Module 21 — Excess Quantity Cases

Review following sections:

Controlled Substances Act regarding Destruction of Excess Quantities

TG Module 21 Material

SOP 16 - Excess Quantity Cases

Module 22 - Trainee Evaluation N/A N/A

Module 22.1 - Competency Samples

Practice samples to be identified by Trainee with assistance as needed

25 Competence samples to be identified by Trainee without assistance.

Module 22.2 - Final Written Exam

Receive Study Guide and Review with Trainer

Take Comprehensive Final Written Exam and Pass with 90% or more

Module 22.3 - Testimony and Mock Trial

Observation of analysts' Testimony throughout the training N/A N/A

Independent Reading

Review following sections:

TG Module 22.3 Material

Lab QASOP — Testimony Monitoring

Practice answering court questions with train

Review of mock video tapes

Mock Trial (in-house) with Review Panel

Receive Training Completion to Begin Receiving Cases

Trainer Signature

Approval Date

Section Manager

Approval Date

Quality Manager

Approval Date

Effective date 02-09-09

HQUST~N POLICE DEPARTMENT CRIME ~ABORATORY
Controlled Substances Trainng Guide MODULE 01
Sub'ect: Ir~trocluction an General Qrien4ation Version 200r

Pa 8 f ~4
TRAINING GUIDELINES FOR NEW EMPLOYEES

- The purpose of this manual is to provide a uniform coordina#ion of #he training of new empoyees to the Houston Police De~artment Crime Laboratory Cor~trolled Substances sec#ion. This manual is in#ended to be used in a format training program that wil~ establish a certain minimum standard of professional competency for the analysis of unknown substances submitted as evidence to the HPD Cr€me Lab.
- Trair~ing is expect#ed to ~ast approximately 5-fi months. The program incltades formal lectures, assigned reading, practical exercises, instrument training and practice, monitored case work, observation of courtroom timony, identification of unknown samples, w~tten examiRations, and a mock . t the end of the training program, the trainee will ha~e the basic skills~ s to perform analyses on the majority of drug cases recei~ec~. r'~~
- The training program for #he Controlled Substa~~ tiec on wi~l be o~erseen by a Training Coordinatot (TC) who will ~e the ubstances L.ab Manager or a designee. Ac#ual training of new empl s w be conducted by a Lab Manager designated Trainer(s) wh ' t defegate certair~ cluties and #~locks of instruction to other analyst ' h approvaf of the section Lab Manager.
- All trainees are expected to e t during the training period. It is the responsibility of the train ob in any iRforma#ion missed due to absence.
- Trainees are exper~# afl material thorough~ly. The time to ask questions is during the tr~n' ~ pr ss.
- Trainees expe ed to keep a personal log of their ac#ivities during training. A weeicly su is to be provided to the Trainer.
- The performance of the trainee will need to be evaluated dunnng the course of the program. This evaluation wilf consist of periodic oral reviews and review of doc~mentat~ort for p~actica~ exercises. The Trainer wilf in turn advise the section Lab Manag~r of the trainee's progress #hroughout the training period.
- A mid-term written exam will be given approximately half way through the training process to inc~ude materiai co~ered to date. A study guide wilf be provided to the trainee which will be reviewed with the Trainer. To pass.the exam the trainee is

expected to answer 90% of the questions correctly. Each question will have the point value clearly indicated. There will be an opportunity to obtain extra credit during the exam.
Effective date 02-09-09

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Controlled Substances Training Guide MODULE One
Subsection: Introduction and General Orientation Version 2006

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After the necessary materials have been covered by the Trainer, each trainee will be expected to identify 25 unknown competency samples. During the identification process, each trainee is expected to work independently. This means that the trainee is not allowed to ask questions about the meaning of any test result nor about the operation of any instrument. The other analysts will be informed when the competency sample analysis begins to ensure that the trainees are left alone to do their analysis.

•

A comprehensive final written exam will be given over information provided in the training guide, during laboratory practical examinations, and in the reading materials. A study guide will be provided to the trainee which will be reviewed with the trainer. To pass the exam the trainee is expected to answer 90% of the questions correctly. Each question will have the point value clearly indicated. There will be an opportunity to obtain extra credit during exam.

•

If any trainee is unable to successfully complete the exams or competency samples, they will be referred to the Director.

•

After successful completion of the written exam and competency samples, trainees will take a Mock Trial.

•

Trainees who successfully complete all parts of the training process including written exams, competency sample mock trial will be issued a memo authorizing them to begin receiving evidence for analysis.

The Trainer is responsible for ensuring all necessary documentation generated during the training process will be maintained in the section's training files.

This includes the trainee's log, reviewed practical exercises, initialed reading lists, notes, competency sample analysis, initialed

syllabus/check completion memo. In addition, a copy of the initialed syllabus/checklist completion memo will be provided to the Quality Assurance Manager.

After the new analyst has successfully completed the training program, there is a period of adjustment from training to full-time analyst. While the training program is designed to provide the new analyst with the information necessary to handle

the majority of evidence submitted for analysis, the new analyst is encouraged to seek the advice of the Trainer, senior analysts, or the section Lab Manager when questions arise.

Following approximately 6 months of active case analysis, the new analyst will perform Technical Reviews of at least 200 case files which will be seconded by the Trainer or another qualified analyst. When the Trainer and section Lab Manager are confident in the ability of the new analyst to perform Technical

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NOIUSTON POLICE DEPARTMENT CRIME LABORATORY
Controlled Substances Training Guide MODULE 01
Subject: Introduction and General Orientation Version 2a09
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Reviews, the new analyst will be authorized to perform such reviews on active case files.

- Since no training program can provide all the skills needed for all types of casework and investigations, it is the responsibility of the trainee to continue his/her training after the formal training period is completed. This involves a good deal of reading about analytical techniques and general background information about drugs and their actions. In addition, in-service schools and meetings will provide new and up-to-date information and skills.

-

It is hoped that each trainee will enjoy the training period and participate in the training process by giving input and adding his/her personal skills to the laboratory. There are two things that all trainees should remember: we are all here to help you learn; and there is no such thing as a d education.
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SKILLS AND KNOWLEDGE QUESTIONNAIRE

The following questions are for the purpose of providing the trainee with an idea of important general chemistry needed to get the most out of the training program. They also provide the Trainer with insight into the knowledge base of the trainee before the program begins.

1. What was your favorite Chemistry class and why?

2. What was your least favorite Chemistry class and why?

3. What pH is considered neutral, acidic, or basic?

4. How do you make a basic solution acidic?

5.

How do you make an acidic solution?

6.

Which of the following techniques are familiar to you and briefly describe your experience with each (class):

TECHNIQUE EXPERIENCE

TLC

MICROSCOPY

GC/FID

GC/MS

IR SPECTROSCOPY

FTIR SPECTROSCOPY

UV/VIS SPECTROSCOPY

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Controlled Substances Training Guide . MODULE 01
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- 7• Give examples of organic solvents?
- 8• Explain the terms soluble and insoluble.
- 9• Explain the terms miscible and immiscible.

10. How would you prepare 100 ml of 3% hydrogen peroxide solution from a 30% stock solution?

~ 1. How would you prepare 200 ml of 5N potassium hydroxide?

(MW KOH = 56g)

12. When preparing a dilute acidic solution from a concentrated acid, do you add acid to water or water to acid?

- 13. What are universal indicators?
 - 14. What is an amine compound? (give an example, general or specific)
 - 15. What is an organic alcohol? (give an example, general or specific)
 - ~6. What is an organic ketone? (give an example, general or specific)
 - 17. What is an organic ether? (give an example, general or specific)
- Effective date 02-09-09

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• What is an organic acid? (give an example, general or specific)

9. What is an inorganic or mineral acid? (give an example)

20. What is an organic ester? (give an example, general or specific)

21. What are cis / trans isomers? (give an example, general or specific)

22. What are enantiomers and diastereomers? (give an example, general or specific)

23. What is the difference between mass and weight?

24. Convert 250 milligrams to grams

25. Convert 2,250 milliliters to liters

26. Convert 4.4 kilograms to pounds. (using 1 kg = 2.2 lb)

27. Convert 5 grams to ounces. (using 1 oz = 30 g)

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HOUSTON POLICE DEPARTMENT CRIME LABORATORY

MODULE 01

Controlled Substances Training Guide

Subject: introduction and General Orientation Version 2U09
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DEPARTMENTAL AND LAB FUNCTIONS

It is important for new employees to the HPD Crime Laboratory to understand the structure of the organization to which they belong and its stated mission. The Trainer will review the various sections of the Crime Laboratory and their functions as well as the organizational structure of the Crime Lab within the HPD and within the City of Houston. The mission statements of both the HPD and of the Crime Laboratory will be discussed during review of the Department's General Orders (GO) and the Lab's Quality Assurance and Standard Operating Procedures (QASOP).

SAFETY TRAINING

Providing employees with proper safety equipment and training is very important to the

City of Houston and to the HPD Crime Laboratory. To in this goal, new employees will receive a Safety Manual, view safety videos, and receive safety equipment.

ACCREDITATION

~

Texas law enacted in 2003 requires crime laboratories to be accredited by the Department of Public Safety by Q9-01-05. The Crime Laboratory was accredited by the American Society of Crime Laboratories, Laboratory Accreditation Board (ASCLD-LAB) in May 2005. Because a certification by a recognized accrediting body, the DPS Director accredited the Department's Laboratory in May 2005 as well. To assist trainees with understanding the accreditation process, Texas Laws regarding regulatory requirements for crime laboratories will be reviewed. The current accrediting body manual will be provided and reviewed with the trainee.

CASEWORK

Throughout the training period, the trainee will be given the opportunity to observe other analysts perform casework. This will help clarify for the trainee the duties of a forensic chemist at the HPD Crime Laboratory.

STUDY GOALS

By whom is the HPD Lab accredited?

•

When did the lab first receive accreditation and when does the current term end?
What are the three types of criteria by which compliance is measured and what

percentage of each criteria must ~e met in order to achieve accreditation by
ASC~D-LAB7
Effective date 02-09-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY
Controlled Substances Training Guide MODU-- 02
Version 2009
Sub'ect: Histo and Control of D of A use pa 8 1 of 4
HISTORY AND CONTROL OF DRUGS OF ABUSE READING LIST

(to be initialed when comple#ed)

1) R. Saferstein, Criminalistics: An Introduct~on to Forensic Science, 7~' Ed, 200'~
Ch. 1 - ^Introduction"
Ch. 9 - "Drugs"

Answer the questions at the end of each chapter

2) Htstory Channel DVDs: "Enemy Within: Drugs and the War to Stop Them"

"Hooked: Illegal Drugs and H They Got That Way"

3) US Dept. of Justice DEA — Drugs of Abuse, 2005 Ed.

4) Amera-Cf~em, Inc. — Drug Identification Bibl 20

"Drugs o# Abuse", p. 1-3.

5) S. Belf, Forensic Chemistrv, 20Q6.

C~. 1 - "Introduction"

6} HPD Academy Cadet Presentat'o a uf on "Narcotics-Laboratory"
Effective date 02-09-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 02
Controlled Substances Training Guide Version 2009
Sub'sect: History and Control of Drugs of Abuse Page 2 of 4
OBJECTIVES

- To familiarize the trainee with background information regarding the history of drug abuse and legal efforts to reduce or control that abuse.
 - To explain the operation of local, state, and federal law enforcement agencies and court systems.
- THE LAW AND DRUGS OF ABUSE
- The following is a summary of various laws enacted within the United States over the years to regulate drugs or respond to drug abuse.

- 1848 — Drugs and Medicine Act
Response to New York College of Pharmacy Philadelphia College of Pharmacology concerning prohibition of sugar called for inspection of drugs and chemicals.
- 1887 — Trade agreement between U.S. and Britain
Neither country was allowed to export to the other.
- 1906 — Pure Food and Drug Act
A drug was considered mislabeled subject to penalties if its label bore any false statements regarding ingredients.
- 1914 — Harrison Narcotic Law
U.S. agrees with others at the International Opium Convention to limit amount of opium for export.
- 1936 — Food, Drug, and Cosmetic Act
Controls opium and coca leaves. Reenacted 1960 by Narcotics Manufacturing Act.
- 1938 — Food, Drug, and Cosmetic Act
Outgrowth of Pure Food and Drug Act. Prohibited interstate movement of adulterated and misbranded drugs. New drugs forbidden until FDA approved New Drugs Application.
- Durham-Humphrey Amendment to FDCA Act
Must have a prescription for certain drugs. Some drugs delineated as over the counter.

Effective date 02-09-09

HQUSTQN PO~ICE DEPARTMENT CRIME LABORATORY MODiJ~E 02
Controlled Substances Training Guide Version 2049
Sub'ect: Hista anc! Control of Or~ s of Abus pa e 3 of 4

Narcotics Manufacturing Act of 1990
Divided drugs into 4 categories
A— Great addictive powers and required tax stamp
B— Lower addictive powers; local prescription could be given.
X—"Exempt" narcotics — only slightly addictive
M — Addictive powers less than "X"

•

Kefauver-Harris Bill or Drug Amendments of 1962
Required the registration of pharmaceutical companies with the FDA
1965 — First Drug Abuse Control Amendment
Controlled some stimulants, barbiturates, and hallucinogens.
Federal Controlled Substances Act of 1970

1.
Created a new category of drug, controlled
2.
Classifies controlled substances into on three criteria
 - a.
Potential for abuse
 - b.
Accepted medical usage
 - c. Likelihood of addictionSchedule I has highest abuse to no medical use, high addictive potential. Schedule V has low potential, high medical usage, lowest addictive potential.

3.
Controlled by DEA, a part of Department of Justice brought into existence in 1973

4.
Defines the method by which substance is controlled.
 - a.
DEA gathering information on the drug's abuse potential
 - b.
DEA requires FDA study medical evaluations on the drug and the procedure ends and no control

5.
Texas Controlled Substance Act of 1993

1.
Contained in Chapter 481 of the Texas Health and Safety Code.
2.
Drugs listed in Penalty Groups 1- 4 set by the Commissioner of Health.
3.
Penalties for possession, delivery, and manufacturing set by the State

Legis~ature.

4.

Also cvntains information on analogues, drug paraphema#ia, etc.

.

Simulated Confrolled Suhstances

Contained in Chapter 482 0# tt~e Texas Heaifh and Safety Code

Dangerous Drugs

Contained En Chapter 483 of the Texas Health and Safety Code.

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HOUSTON POLICE DEPARTMENT CRIME LABORATORY
Controlled Substances Training Guide MODULE 42
Version 2/10/09
Subject: History and Control of Drugs of Abuse page 4 of 4

- Abusable Volatile Chemicals
Contained in Chapter 485 of the Texas Health and Safety Code
• Over-the-Counter Sales of Ephedrine, Pseudoephedrine, and
Norpseudoephedrine
Contained in Chapter 486 of the Texas Health and Safety Code

LAW ENFORCEMENT AGENCIES AND THE COURT SYSTEM

The Houston metropolitan area consists of neighboring and overlapping law enforcement agencies and legal jurisdictions. Most of the evidence received by the HPD Crime Lab is submitted by HPD officers recovered from within the city limits. However, the Crime Lab will receive multi jurisdictional evidence as the result of task force seizures or inter-agency cooperation. Evidence recovered within Harris County but outside of Houston's city limits will be sent to the Harris County Medical Examiners Office for processing. Some evidence submitted to the Lab will come from the portion of Houston that lies within Fort Bend County's County there is also the Pasadena Crime Lab serving the City of Pasadena Texas Department of Public Safety Lab serving surrounding counties.

An analyst with the HPD Crime Lab may testify in various jurisdictions. Within the City of Houston there are county courts (dealing primarily with misdemeanor offenses), State of Texas dealing primarily with felony offenses), and United States Federal courts (dealing with Federal or interstate offenses). In addition, analysts will sometimes go to Federal courts located in other parts of Texas or across the United States.

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The trainee will be provided current copies of the Texas Health and Safety Codes Chapters 481-486 in order to sources of information regarding the Federal Controlled Substances Act. This will be reviewed with the trainee who will be required to be familiar with their texts.  
STUDY GOALS

- Understand the basis of the Federal Controlled Substances Act and the Scheduling of substances at the Federal Level (i.e. How many schedules are there and what is the basis for placing a particular substance into one of the schedules?)  
• Understand the difference between Controlled Substances, Dangerous Drugs, and Over-the-counter substances in Texas.  
Be able to identify the Penalty Group for various substances.

Effective date 02-09-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY  
Controlled Substances Training Guide MODULE 03  
Version 2409

CHEMICAL CLASSIFICATION OF DRUGS READING LIST  
(to be initiated when completed)

1) R. Saferstein, Criminalistics: An Introduction to Forensic Science, 7<sup>th</sup> Ed, 2001  
Ch. 2 - "The Crime Scene"  
Ch. 3 - "Physical Evidence"  
Ch. 4 - "Physical Properties" pp. 87-97.  
Ch. 5 - "Organic Analysis"  
Answer the questions at the end of each chapter

2) A.C. Moffatt editor, Clarke's Analysis of Drugs and Poisons, 3<sup>rd</sup> Edition, 2004.  
Ch. 2 — "Drugs of Abuse"  
3) Amera-Chem, inc. — Drug Identification Bibliography 2008  
"Drugs of Abuse", p. 274-707.

4) S. Bell, Forensic Chemistry, 2006.  
Ch. 5 - "Instrumentation"  
Ch. 6 - "Overview of Drugology"  
Ch. 7 • "Forensic Drug Analysis"  
Ch. 8 - "Forensic Analytical Chemistry of Drugs"

5) J. F. Casale, et. al. "Forensic Cocaine Impurities...from cis- and trans-cinnamoylcocaine"  
Journal of Forensic Sciences, 52(4), July 2007, pp.  
560-566.

6) K. K. Rasmussen. Cocaine. Marijuana, Designer Drugs, Chemistry,  
Pharmacology and Behavior, 1999.  
Ch. "Designer Drugs: An Overview"  
Ch. "Substituted Amphetamine Controlled Substance Analogues"

7) CND Analytical: Series of Analytical Profiles - Introductions from the following:  
Forensic and Analytical Chemistry of Clandestine Phenethylamines  
Amphetamines and related phenethylamines  
Substituted 3,4-methylenedioxyamphetamines:

Designer Drugs related to MDA vol. 1 and vol. 2  
Barbiturates and other Depressants  
Benzodiazepines  
Cocaine, Local Anesthetics and Common Diuretics  
Hallucinogens



Narcotic A~a~gesfcs

Effective date 02-09-09

NOUSTON POLICE DEPARTMENT GR1ME LABORATORY MODULE 03  
Controlled Substances Training Guide

Version 2009

Subject: Chemical Classification of Drugs

Page 2 of 11

## OBJECTIVES

- To familiarize the trainee with different classes of drugs of abuse (based on effects or molecular structure).
- To familiarize the trainee with simple pharmacology of the major classes of drugs.
- To familiarize the trainee with the molecular structures of the most commonly abused drugs and substances of interest.
- To introduce the trainee to various techniques of analysis in relation to chemical structures (for example, secondary amines turn blue in the ferricyanide spot test and amphetamines give a "three-finger" UV absorption reading).

## DISCUSSION

- The Trainer will use the charts and structures following pages to discuss the classification of substances based on effects (uses) and point out structural similarities and differences in classes.
  - As these substances are discussed, the Trainer will introduce various analytical techniques and relate the functional groups present in the molecular structures.
  - Other topics such as street manufacture, precursors, metabolites, isomers, acidic vs. basic drugs, primary, secondary, and tertiary amines, etc. may be included in discussion.
- Effective date 02-09-09

NOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 03  
Controlled Substances Training Guide  
Subject: Chemical Classification of Drugs Version 2009

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~EiUG C~~~~~y~i~i~j~~ AN~3 S~RUGTURES  
AMPHETAMINE AND RELATED COMPOUNDS

$\text{H}_2\text{C}-\text{CH}_2-\text{CH}_3$   
 $\sim \text{CH}_2-\text{CH}-\text{CH}_3 \sim \text{CH}_2-\text{CH}-\text{CH}_3 \sim \text{CH}_2-\text{C}-\text{CH}_3$   
| ~ ~ !

/// CH<sub>3</sub>

Amphetamine Methamphetamine Phentermine

$\text{H}-\text{CH}_2-\text{CH}_3$

f ~ CH<sub>2</sub>•CH—CH<sub>3</sub> ~ CH<sub>2</sub>-C--C ~ CH<sub>3</sub>

|

~ i / NH<sub>2</sub>

cF~

Fenfluramine Phentermine Methylamine

~ ~

OH

z ~ = . . H-CH<sub>3</sub> H`C /NH-CH<sub>3</sub>

C~C ~ ~C

H<sub>3</sub> | H: ~CH<sub>3</sub>

H` \

~ ~ ~

` f-Ephedrine d-Pseudoephedrine

NH-CH<sub>3</sub>

iH—CH<sub>2</sub>•CH

-CH<sub>3</sub> ~ CH<sub>2</sub>-CH-CH<sub>3</sub> ~ CH<sub>2</sub>-CH—CH<sub>3</sub>

||

~ .

0 0

! H~

~H<sub>2</sub> 2

3,4-Methylenedioxy 3,4-Methylenedioxy 3,4-Methylenedioxy

Amphetamine Methamphetamine N-Ethylamphetamine

Effective date 02-09-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 43  
Controlled Substances Training Guide Version 2009  
Subject: Chemical Classification of Drugs Page 4 of 11

Various opiates

Codeine Morphine Heroin (Diacetylmorphine)  
Naloxone  
Hydrocodone Moxycodone

Dextromethorphan Acetylcodeine  
Noscapine Papaverine Thebaine  
Cough

~

5 BH5 i 3

~ H2 C6H3

CH<sub>3</sub>—CH<sub>2</sub>—O—C—CH—CH<sub>2</sub>—N(CH<sub>3</sub>)<sub>2</sub> C<sub>6</sub>H<sub>5</sub>—CH<sub>2</sub>—CH<sub>2</sub>—CH<sub>2</sub>—CH<sub>2</sub>—N(CH<sub>3</sub>)<sub>2</sub>

~ 5 H5 O C<sub>6</sub>H<sub>5</sub>

Dextropropoxyphene Methadone  
Effective date D2-09-09

HOI151'ON POLICE pEPARTMENT CRfME LABORATORY

MODUL~ 03

Cor~tralled Substances Training Guide

Version 2009

Sub'ect: Chemical Classifica#fon of Dru s

pa 5 of T

~~~~~iE; L~C~iL Ei~VEST~iETiCS ~-ND REL~~`ED COMPOUNDS

N,CH~ lo~~~3 /~H~ ~ ~cH3

~N ~N _ O

a--C~--CF~CH~{~

--O~II \ I

N a ~--i

I-Cocaine d-Cocaine Cinnamoylcocaine

~

Benzocaine Lidocaine Tetracaine

iCs~~ ~Ca~s

NFEZ \ / I~—O—CHz—CH2—CHZ—N

NH2 \ ~ I~—~—CH2--CH2—N~

p CzM5 O \C4~a

Procaine Butacaine

Effect~ve date 02-09-09

~ --- ~ - - - - - ~ ---HOUSTON
PO~ICE DEPARTMENT CRIME LABORATORY
Controlled SubstaRCes Training Guide MODULE 03

Version 2009
Sub'ect: Chemical Classi~cation of Dr~ s Pa e 6 of 11

BAiRBiTUF~-TES
4

NH O NH O ~N~i O

~ NH NH CHZ-CH3 NH ~Hz-CH3

CsHS C~iZ-CHZ-~H-CHa

O O O CH3

Barbituric Acid Phenobarbital Amobarbitaf

O NH O ~~~~ ~ O NH O

~ NH CH2-CH=CH2 NH CH2-CH~CH7 ~ CH2-CH3

NH

CHz-CH-CH3 C~N-CHZ CHZ-CN3 CH-CH2-CHp--CH

~ CF~3 ~

O CH3 O CH~

Butalbital Secobarbit Pentobarbital

ANALG f

COOH H

\ O--li--CH3

~ O I \

//

II

N Ff—C--CH3

Aspirin Acetaminophen

~~~

CH<sub>3</sub> H<sub>3</sub> C H--COOH

~ ~ q \_

jCH—CH<sub>2</sub> \ / CH-COOH

CH<sub>3</sub>

~ ~

Eb~prafen Ketoprofen

ABUSABLE VOLATILE CHEMICALS

~3 CH<sub>3</sub>

CF~CF+~--CKz CH<sub>2</sub>—O—I~O jCH-CH<sub>2</sub>-O—N=O

CH<sub>3</sub>

~ `

/

Toluene Butyl Nitrite Isobutyl Nitrite  
Effective date 02-OS-09

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Version 2009

Subject: Chemical Classification of Drugs Page 7 of 11

BENZODIAZEPINES

Diazepam Alprazolam Lorazepam

or

Flunitrazepam Clonazepam

CN3~~

N \ N ~ H O N

/ + / ~ COOH

Cl \ Cl \ ~N

~ Cl ~

\ ~ \ ~

Triazolam Clonazepam Temazepam

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Controlled Substances Training Guide Version 20Q5  
Subject: Chemical Classification of Drugs p. 8 of 11

RI-O-biazines are tricyclic antidepressants  
N-CH<sub>2</sub>-CH<sub>2</sub>-N(CH<sub>3</sub>)<sub>2</sub>

~~~~~

~ S ~

Promethazine Promazine

~~~~~3 P

IVH<sub>2</sub>-C-C-iz ---CH<sub>2</sub>-C-NH<sub>2</sub>

CNq-CH<sub>2</sub>-CH<sub>3</sub>

Meprobamate Metoprolol

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Controlled Substances Training Guide MODULE 03  
Subject: Chemical Classification of Drugs Version 2009

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~M~V VII VVG IY7

N ~ NH—CH<sub>3</sub>

~ ~

N

E /

/ \

Phencyclidine 1-Piperidinocyclohexane Ketamine

Carbonitrile

{PCP intermediate}

~N

~ \ ~

/ ~CH<sub>a</sub>

CH<sub>3</sub>-Cl-iz Pf

OH ~CH<sub>3</sub>

,CNZCH<sub>3</sub>

~CHZCH<sub>3</sub>

H

Lysergic Acid Diethylamide

{LSD}

CANNABINOIDS

Cannabidiol acid (CBDA) Cannabidiol (CBD)

,f S1

Cannabinof (CBN) 9,10-Tetrahydrocannabir~oi (THC)  
EfFective date 02-09-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MQDUL~ 03  
Controlled Substances Training Guide Version 2409  
Subject: Chemical Classification of Drugs Page 10 of 11

OTHEi~ C~iYrMON IDE~UG~ Aiii~'i SUo~~i AFi~~~ ^v~ ii~i~i Er~E~T  
a~ ~ ~3 ~  
ocM,/ CH2-CH-CHz OH CH3 .N ~ N ~ ~ ~ \ ~ II ~NHZ

O i O  
\ ~ CH3  
Guaifenesin Caffeine Nicotinamide

~  
~~ } H NH—Cl  
~ \ / O ~ C`~2  
N~ H-CHz•CM2-N(CH3)2 I '  
I ~  
Chlorpheniramine 1 Phenylephrine  
Sildenafil {Viagra}  
Fluoxetine (Prozac}  
O O O  
OH-CH2-CHZ-CH2-C—OH ~~ OH-CHz CH~-CHZ-CHz ~H

gamma-Hydroxybutyric Acid gamma-Butyrolactone 1,4-Butanediol  
Effective date 02-09-09

## HOUSTON POLICE DEPARTMENT CRIME LABORATORY

Controlled Substances Training Guide MODULE 03

Sub'sect: Chemical Classification of Drugs Version 2009

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TRAINING GLIDE EIRQL-Q-RA-HS

The trainees will be provided with a Training Guide Monograph Appendix which is intended to give a quick reference for some of the more commonly encountered controlled substances, dangerous drugs, and other substances of interest. This document is updated periodically and can be a useful reference during and after training.

### STUDY GOALS

- Understand the terms Narcotic, Depressant, Stimulant, and Hallucinogen in relation to the effect of a substance on the body and be able to identify the effect on the body of various substances.
- Be able to match the chemical name for various substances to the provided structures.
- Understand the term opium, its source, how it is used, the principle alkaloids present and their relative abundances. Understand the term narcotic as well as be able to identify various narcotics naturally-occurring, semi-synthetic, or synthetic.

Effective date 02-09-09

H4USTON PO~ICE DEPARTMENT CRIME LABORATORY  
Controlled Substances 7`ralning G~ide MODULE 04  
Sub~ect: Pharmaceutical Identifiea#ion of Dru s Version 200~

pa e 1 of 3

PHARMACEUTICAL IDENTIFIcAT~ON READING LIST

{to be ini#iafed when comple#ed)

~'~ ) A.C. Moffat editor, Gla~fce's Anal sis of Dnl s and Poisons, 3rd Edition, 2004.

Ch. 17 — "Solid Dosage Ip"

2) Amera-Chem, Inc. — Drug Identifrcation Bi61e, 200\$ Ed.

"Pharmaceutical Id", p. 4 — 273.

Effectl~e date 02-09-09

HQUSTON POLICE pEPARTMENT CRIME LABORATORY  
Cntralled Substances Training Gulde MODUL~ 04  
Svb'ect: pharmaceutical Identffication of Dru s Version 2009

Pa e 2 of 3

## OBJECTIVE

To familiarize #Fte trainee with various methods of identifying pharmaceutical products by their markings or labels.

## DISCUSSION

Most pharmaceutical products (whether tablets, capsules, or liquid containers) have markings (logos) or labels which are intended to identify both the manufacturer and the ingredients or contents. Identification of these products involves the use of reference materials to visually match an unknown pharmaceutical prepared dosage form.

+

### National Drug Code Directory

This is referred to as the NDC number, and it is required all companies and their products be registered. This number is found on the original pharmaceutical manufacturer's containers.

•

### Physicians Desk Reference

~.

Drugs listed by chemical (generic), product name, product class, and manufacturer.

2.

Updated on a yearly basis.

3.

Contains picture index of number of dosage forms for visual ID.

4.

Companies must have products advertised in the PDR. Most generic manufacturers listed.

5.

The manufacturer contacted directly for information. The phone numbers and addresses are listed in the front of the PDR.

•

### Drug Identification

1.

Identification indexed in alphabetical order for the imprint on the tablet or capsule.

2.

Pictorial representations for selected tablets and capsules.

3. Useful information on various drugs.

### DEA Logo Search

Tablets and capsules can be searched by their markings, appearance,

manufacturer, etc. #n a computer database. The menu that comes up is selfexpfanatory. The database is updated periodically and ~is available from the D EA.

#### Amera-Chem Logo Search

This is another computer database search routine that may be purchased thro~gh tF~e Dn,g Identification Bible website ([www.druQidbible.com](http://www.druQidbible.com),. It is available In CD-ROM format and can generate cofor prin#outs for most ot tf~e products.

Effective date 02-09-09



HOUSTON POLICE DEPARTMENT CRIMINAL LABORATORY

Controlled Substances Training Guide MODULE 04

Subject: Pharmaceutical Identification of Drugs Version 2009

Page 3 of 3

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Internet Links such as [www.drugs.com](http://www.drugs.com) or search engines like Google or Yahoo can also be useful in identifying products.

- 

Poison Control Center

1. Staffed 24 hours daily to answer questions from the public.
2. Texas Poison Control Center: UT Med Branch, Galveston 800-222-2222
3. This should only be used as a last resort.

#### PRACTICAL EXERCISES

The trainee will be provided with a Pharmaceutical Identification Practice Worksheet

- 

The information requested  
(ingredient, and control  
status information)

+ information

DOCUMENTATION

The Trainer will labus/Checklist)

and will provide information Sheet

documentation of

#### STUDY GOALS

Be able to identify all identification  
and know their names

Effective date 02-09-09

CHEMICAL SCREENING 1 SP4T TESTS READING LIST  
(to be initiated when completed)

- 1) A.C. Moffat editor, Clarke's Isolation and Identification of Drugs, 2<sup>nd</sup> Edition, 1986. "Color Tests", p. 28 — 147.
- 2) A.C. Moffat editor, Clarke's Analysis of Drugs and Poisons, 3<sup>rd</sup> Edition, 2004. Ch. 9 — "Colour Tests"
- 3) S. H. Johns, et. al. "Spot Tests: A Color Chart Reference for Forensic Chemists", Journal of Forensic Sciences, 24 (1979), pp. 631-649.
- 4) L. J. Scott, "Specific Field Test for Cocaine", 73, pp. 179-182.
- 5) C. L. Rucker, "Chemical Screening and Techniques for Funitrazepam", Microgram, 31(7), Jul 2005.
- 6) A. S. Garrett, "The Weber Test: A Color Test for the Presence of Psilocin in Mushrooms", SWAFS Journal, 1993, pp. 44-45.
- 7) W.J. Stall, "The Cobalt Nitrate", Microgram, 13(3), March 1980, pp. 40-43.
- 8) A. L. Deakin, "A Test for the Acidified Cobalt Thiocyanate Test for Cocaine", Journal, 1(1-2), January - June 2003, pp. 40-54.
- 9) J. A. Moore of GHB for FTIR Analysis and a New Color Test for Ganaxone (GBL), Microgram, 32(8), August 1999, pp.
- 10) D. M. Chiong, et. al. "The Analysis and Identification of Steroids", Journal of Forensic Sciences, 37(2), March 1992, pp 488-502.
- 11) J. A. Morris, "Modified Cobalt Thiocyanate Presumptive Color Test for Ketamine Hydrochloride", Journal of Forensic Sciences, 52(1), January 2007, pp. 84-87.
- 12) M. Sarwar, "A New, Highly Specific Color Test for Ketamine", Microgram Journal, 4(1-4), January - December 2006, pp. 24-28.

Effective date 02-09-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE Q5  
Controlled Substances Training Guide  
Sub'sect: Chemical Screening Tests Version 2009

Page 2 of  
OBJECTIVE

- To familiarize the trainee with the preparation, quality control, storage, and proper handling procedures of chemical screening test reagents (also known as spot tests or color tests).

- To make the trainee proficient in the use of chemical screening tests.

- To make the trainee aware of the advantages, disadvantages, and limitations of chemical screening tests.

To familiarize the trainee with the theory of chemical screening tests.

#### DISCUSSION

The Trainer will review appropriate sections of the ~ Syllabus/Checklist) for performing chemical screening tests and for reaction control procedures. The Trainer will also discuss the verification of standards by the Controlled Substances Section and documentation in the Standard U

#### PRACTICAL EXERCISES

- The Trainer will assist the trainee in preparing reagent bottles for his/her work area needed for performing the screening tests.

- The trainee will receive a list of practice samples for performing chemical screening tests.

- The Trainer will state how to perform chemical screening tests.

- Once the trainee has completed the practice worksheet it will be reviewed with the Trainer.

#### DOCUMENTATION

The Trainer will review the proper Examination Sheet documentation for chemical screening test results. This will include documentation of quality checks for infrequently used reagents.

Effective date 02-09-09

## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 05

Controlled Substances Training Guide Version 20a9

Subject: Chemical Screening Tests Aa e 3 of

STUDY GOALS

- Understand the reagent quality control system used by the Controlled Substances Section. Identify various reagents used for spot tests as frequently used or infrequently used.

- 

Understand how to perform various spot tests and the expected results for commonly encountered substances.

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HOUSTON POLICE DEPARTMENT CRIME LABORATORY  
Controlled Substances Training Guide MODULE 06  
Subject: Microcrystalline Test Version 2409

Page 1 of 5  
MICROCRYSTALLINE TESTS READING LIST  
(to be initiated when completed)

1) R. Saferstein, Criminalistics: An Introduction to Forensic Science, 7th Ed, 2001.  
Ch. 7 - "The Microscope"  
Answer the questions at the end of the chapter

2) E.G.C. Clarke editor, Isolation and Identification, of Drugs, Volume 1, 1978.  
"Microcrystal Tests", pp. 135-141. ~ ~ ~

3) J. Swiatko, et. al. "Further Studies on Spot Tests and Microcrystal Tests for  
Identification of Cocaine", Journal of Forensic Science 48(3), May 2003,  
pp. 581-585.

4) S. Bell and R. Hanes, "A Microchemical presumptive Testing of  
Controlled Substances", Journal of Forensic Sciences, 52(4), July 2007,  
PP. 884-888. ~

5) HPD in-house Microcrystalline Proficiency and Control

~

Effective date 02-09-09

## HOUSTON POLICE DEPARTMENT CRIME LABORATORY

### Controlled Substances Training Guide MODULE 6

#### Subject: Microcrystalline Tests Version 2009

Page 2 of 5

#### OBJECTIVE

- To familiarize the trainee with the preparation, quality control, storage, and proper handling procedures of microcrystalline test reagents.
- To familiarize the trainee with microcrystalline tests for various substances.
- To make the trainee aware of the advantages, disadvantages, and limitations of microcrystalline tests.
- To familiarize the trainee with the theory of microcrystalline tests.

#### HISTORY OF MICROCRYSTALLINE TESTS

Since the microscope was invented and produced on a scale, its use began

to spread throughout the scientific community. Naturalists used it to

investigate naturally occurring crystals and Descriptions of these

observations soon followed in books published in the 1700's.

As the science of chemistry became systematic, microscopic examinations

began to include observations of reactions, as identification of various isolation

products from experimental procedures. These early studies involved coiled

products rather than specific crystal reactions. All through the 1800's, this basic

investigation and observation of natural occurring substances formed the major part of the literature.

It was the discovery of the polarizing microscope in 1811 that

opened up the field to study. Chemists and mineralogists began to describe

the crystals in terms of their responses to the polarized lights (extinction, dichroism,

refractions, etc.). The instrument was improved (in 1819 with Brewster's analyzer; in

1828 with Nicol's prism, more distinction between the naturally occurring crystals could be made. In the 1830's, toxicologists began to use the polarizing microscope to distinguish between alkaloids they discovered in poison cases. Reactions of these alkaloids, especially quinine and strychnine, with specific reagents were developed during the 1840's and 1850's.

The first specific crystal test was performed in the early 1840's and is attributed to Herapath. He identified quinine in an unknown sample by reacting it with an iodosulfate solution to give quinine-iodosulfate crystals. 1859 saw the publication of several microcrystal tests for strychnine, one for cyanide, and others.

During the 1860's microcrystal tests were extensively developed. Three different texts were published that covered microcrystal identification of compounds. These early texts reported mainly on the naturally occurring plant alkaloids or inorganic poisons. As time passed



## HOUSTON POLICE DEPARTMENT CRIME LABORATORY

### Controlled Substances Training Guide MODULE 06

Subject: Microcrystalline Tests Version 2409

Page 3 of 5

the field expanded, more organic compounds were tested for their reactions to crystal-forming reagents. By the 1980's, several books on microcrystal tests had been published covering a wide variety of chemicals.

Investigation and expansion of microcrystalline testing to other organic and inorganic compounds continued into the early part of the 20th century. At that time, interest in microcrystalline tests waned as instrumental analyses were developed. Today, there are those who feel that the chemical reactions observed under the microscope are "less precise" than the graphs and tables obtained from an instrument.

### GENERAL INFORMATION

The purpose of microcrystalline tests is to identify small quantities of chemical substances using a polarizing microscope. The polarizing microscope is preferred in order to observe other crystal characteristics, such as birefringence (splitting of a light beam into two components, which travel at different velocities, producing colored crystals), dichroism (property of some crystalline materials of transmitting one of two plane polarized components of transmitted light more than the other), etc. General crystal formation and size can be observed using a polarizing microscope.

A microcrystalline test is a chemical method of identification using chemical precipitation tests, in which the polarizing microscope is used to observe and distinguish the different kinds of crystals formed, is useful for compounds containing basic nitrogen. By using different reagents, it is possible to extend precipitation and crystal tests to all types of compounds which contain nitrogen atoms and as any basic qualities, even

though the molecule as a whole may be neutral or acidic.

The advantages of crystalline tests include simplicity, directness,

convenience, speed, and small amount of material required. One disadvantage is the occasional difficulty in obtaining an exact match between sample and standard. This difficulty can be from

(1)

the presence of sample impurities, which can lead to the formation of deformed and irregular crystals,

(2) polymorphism (the property of crystallizing in two or more forms with distinct structures; can be caused by different conditions of temperature, pH, etc), or

(3) differences based on concentration.

There are two basic uses of microcrystalline tests. The first is the identification of the compound itself. This is carried out by using the same reagent(s), under identical conditions, to compare the results of an unknown to those of a standard reference compound. The second is the identification of the optical isomer of a compound (i.e., its  
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## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 06

Controlled Substances Training Guide Version 20Q9

Subject: Microcrystalline Test Panel 4 f

presence as the d- or l-enantiomer or as a 50:50 racemate. This may be necessary for the following reasons:

- {1 } Some state or federal statutes may specify that one enantiomer is controlled while the other is not, or the penalties may differ between the two enantiomers;
- (2) To assist officers with information about clandestine lab methods being used; or
- (3) To identify isomers in proficiency examinations to obtain possible synthesis information.

Enantiomer identification by microcrystalline testing is not very widely used any longer. More commonly, labs will perform derivatizations followed by gas chromatography to identify different enantiomers.

Some general examples of the various terms used in describing crystals are given below. These examples are not necessarily exact; different forms can merge imperceptibly into one another.

### TESTIMONY ON MICROCRYSTALLINE TESTS

The advent of instrumentation in the laboratory has led to a decrease

in the use of microcrystalline tests. Although these tests are precise and extremely sensitive, their simplicity leads some to question their accuracy. This prejudice is carried over into the courtroom especially by the defense attorneys.

To counter this prejudice, the analyst must be confident in the results of these tests. Before testifying, review all notes on the analysis. Be prepared to justify microcrystalline tests if the case sample contains an adulterant or diluent that could interfere with the tests.

Questions on the specificity and sensitivity of the crystal tests used can be found in Clarke (Volume 1 only). It gives the sensitivity of the tests in 1:X form. For example, the test will give crystals if there is one volume of the substance in up to X volume of the mixture. The sensitivity of these tests varies quite a bit. A generalized knowledge of these values is good to have on hand to answer such questions.

The specificity of these tests has been proven through testing of similar compounds. The unique molecular structure of a substance determines the shape of the resultant crystals.

Microcrystalline tests were developed for the agricultural testing procedures required by the FDA in the early 30's and 40's. The defense might try to bring up the fact that these are "old-style" tests. Chemistry and the physical laws are still valid; the reactions are the same; therefore, the same predictable and reliable results are obtained. Confidence in your procedures, the best defense against any attack, is

built through literature research and extensive practice with standards. Confidenc~ built  
Effective date 02-09-09

## HOUSTON POLICE DEPARTMENT CRIME LABORATORY

Controlled Substances Training Guide MOU-E 08

Subject: Microcrystalline Tests Version 2009

Prepared by

on this broad base cannot be shaken no matter what type of attack is mounted by the

defense.

### DISCUSSION

The Trainer will review appropriate sections of the CS-SOP (see Syllabus Checklist) for performing microcrystalline tests and for reagent quality control procedures.

### PRACTICAL EXERCISES

The trainee will receive a list of practice samples for performing microcrystalline

tests.

- 

The Trainer will demonstrate the proper use of the microscopes available in the Controlled Substances Section.

- 

The Trainer will demonstrate how to perform crystalline techniques including the direct addition of reagents and the drop technique.

Once the trainee has completed the worksheet it will be reviewed with the Trainer.

### DOCUMENTATION

The Trainer will review the proper station SF sheet documentation for microcrystalline tests.

### STUDY GOALS

- 

Understand the reagent quality control system used by the Controlled Substances Section. Identify various reagents used for microcrystalline tests as frequently used or infrequently used.

Understand how to perform various microcrystalline tests and the expected results for commonly encountered substances.

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HOUSTON POLICE DEPARTMENT CRIME LABORATORY  
Controlled Substances Training Guide MODULE 06  
Subject: Microcrystalline Test T Version 2009

Page 1 of 5  
MICROCRYSTALLINE TESTS READING LIST  
(to be initiated when completed)

1) R. Saferstein, Criminalistics: An Introduction to Forensic Science, 7th Ed, 2001.  
Ch. 7 - "The Microscope"  
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3) J. Swiatko, et. al. "Further Studies on Spot Tests and Microcrystal Tests for Identification of Cocaine", Journal of Forensic Science 48(3), May 2003, PP. 581-585.

4) S. Bell and R. Hayes, "A Microfluidic Electrode Resumptive Testing of Controlled Substances", Journal of Forensic Sciences, 52(4), July 2007, PP. 884-888.

5) HPD In-house Microcrystalline  
Effective date 02-OS-09

Controlled Substances Training Guide MODULE 06

Subject: Microcrystalline Tests Version 2009

Page 2 of 5

OBJECTIVE

- To familiarize the trainee with the preparation, quality control, storage, and proper handling procedures of microcrystalline test reagents.
- To familiarize the trainee with microcrystalline tests for various substances.
- To make the trainee aware of the advantages, disadvantages, and limitations of microcrystalline tests.

- To familiarize the trainee with the theory of microcrystalline tests.

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products from experimental procedures. In these early studies involved colored

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investigation and observation of naturally occurring substances formed the major part of the literature.

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the crystals in terms of their responses to the polarized lights {extinction, dichroism,

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## HOUSTON POLICE DEPARTMENT CRIME LABORATORY

Controlled Substances Training Guide MODULE 06

Subject: Microcrystalline Tests Version 24a9

Page 3 of 5

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The purpose of microcrystalline tests is to identify small quantities of chemical substances using a polarizing microscope. The polarizing microscope is preferred in order to observe other crystal characteristics, such as birefringence (splitting of a light beam into two components, which travel at different velocities, producing colored crystals), dichroism (property of some crystals of two plane

polarized components or transmitted light more so than the other), etc. General crystal formation and size can be observed using a compound microscope.

A microcrystalline test is a chemical identification using chemical precipitation tests, in which the polarizing microscope is used to observe and distinguish the different kinds of crystals formed. It is useful for compounds containing basic nitrogen. By using different reagents, it is possible to extend precipitation and crystal tests to all types of compounds in which nitrogen atom has any basic qualities, even

though the molecule as a whole may be neutral or acidic.

The advantages of crystalline tests include simplicity, directness, convenience, speed, and small amount of material required. One disadvantage is the occasional difficulty in obtaining an exact match between sample and standard.

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and irregular crystals,

(2) polymorphism (the property of crystallizing in two or more forms with distinct structures; can be caused by different conditions of temperature, pH, etc), or

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There are two basic uses of microcrystalline tests. The first is the identification of the compound itself. This is carried out by using the same reagent(s), under identical conditions, to compare the results of an Unknown to those of a standard reference compound. The second is the identification of the optical isomer of a compound (i.e., its  
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1. The purpose of this module is to provide the student with the information necessary to perform the microcrystalline test. The student should be able to identify the various forms of controlled substances and the microcrystalline test results. The student should be able to identify the various forms of controlled substances and the microcrystalline test results. The student should be able to identify the various forms of controlled substances and the microcrystalline test results.

the following reasons:

(1)

Some state or federal statutes may specify that one enantiomer is controlled while the other is not, or the penalties may differ between the two enantiomers;

(2) To assist officers with information about clandestine lab methods being used; or

(3) To identify isomers in proficiency examinations to obtain possible synthesis information.

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To counter this prejudice, a lawyer must be confident in the results of these tests. Before testifying, remember to take notes on the analysis. Be prepared to justify microcrystalline tests if the case sample contained an adulterant or diluent that could interfere with the tests.

Questions on the efficacy and sensitivity of the crystal tests used can be found in Clarke (Volume 1) which gives the sensitivity of the tests in 1:X form. For example, the test will give a crystal if there is one volume of the substance in up to X volume of the mixture. The sensitivity of these tests varies quite a bit. A generalized knowledge of these values is good to have on hand to answer such questions.

The specificity of these tests has been proven through testing of similar compounds. The unique molecular structure of a substance determines the shape of the resultant crystals.

Microcrystalline tests were developed for the agricultural testing procedures required by the FDA in the early 30's and 40's. The defense might try to bring up the fact that these are "old-style" tests. Chemistry and the physical laws are still valid; the

reactions are the same; therefore, the same predictable and reliable results are obtained. Confidence in your procedures, the best defense against any attack, is built through literature research and extensive practice with standards. Confidence built  
Effective date 02-09-09

# HOUSTON POLICE DEPARTMENT CRIME LABORATORY

## MODULE 06

Controlled Substances Training Guide  
Version 2009

Subject: Microcrystalline Tests ~ Part of 5  
on this broad base can't be shaken no matter what type of attack is mounted by the  
defense.

### DISCUSSION

The Trainer will review appropriate sections of the CS-SOP (see Syllabus Checklist) for  
performing microcrystalline tests and for reagent quality control procedures.

### PRACTICAL EXERCISES

- The trainee will receive a list of practice samples for performing microcrystalline  
tests.

- The Trainer will demonstrate the proper use of the microscopes available in the  
Controlled Substances Section.

- The Trainer will demonstrate how to perform crystalline techniques  
including the direct addition of reagents and the drop technique.

- Once the trainee has completed the practical sheet it will be reviewed with  
the Trainer. DOCUMENTATION

The Trainer will review the proper annotation Sheet documentation for microcrystalline  
tests.

### STUDY GOALS

- Understand the entire quality control system used by the Controlled  
Substances Section. Identify various reagents used for microcrystalline tests as  
frequently used or infrequently used.

- Understand how to perform various microcrystalline tests and the expected  
results for commonly encountered substances.

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HOUSTON POLICE DEPARTMENT CRIME LABORATORY  
Controlled Substances Training Guide MODULE 07  
Subject: Midterm Written Exam Version 2009

Purpose  
OBJECTIVE

The purpose of the written midterm examination is to provide the trainee with an opportunity to demonstrate technical knowledge related to the analysis of controlled substances, dangerous drugs, and other chemical substances as covered in Modules 01-06 (Introductory Material thru Microcrystalline Testing).

MIDTERM EXAMINATION STUDY GUIDE

Accreditation:

By whom is the HPD lab accredited?  
When did the lab first receive accreditation and when will the current term end?

- What are the three types of criteria by which labs are measured and what percentage of each criteria must be met in order to achieve accreditation by ASCLD-LAB?

Detecting Controlled Policies:

- Understand the basis of the Controlled Substances Act and the Scheduling of substances at the federal level (i.e. How many schedules are there and what is the basis for placing a particular substance into one of the schedules?)

- Understand the difference between Controlled Substances, Dangerous Drugs, and Over-the-counter substances in Texas.

- Be able to identify the Penalty Group for various substances (see table).

Drug Classifications and Effects:

Understand the terms Narcotic, Depressant, Stimulant, and Hallucinogen in relation to the effect of a substance on the body and be able to identify the effect

on the body of various substances (see table).

Be able to match the chemical name for various substances with the provided structures (see table).

- Understand the term opium, its source, how it is abused, the principal alkaloids present and their relative abundances. Understand the term narcotic as well as be able to identify various narcotics as naturally-occurring, semi-synthetic, or synthetic.

Effect of various substances on the body

## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 07

Controlled Substances Training Guide Version 2009

Subject: Midterm Written Exam - Q 2 Qf

- 

Before the exam, you will be given a list of 85 substances to identify using the PDR, DEA Logo Search, ACS, and DIB).

Spot Tests 1 Screening Tests:

- 

Understand the reagent quality control system used by the Controlled Substances Section. Identify various reagents used for spot tests as frequently used or infrequently used.

- 

Understand how to perform various spot tests and the expected results for commonly encountered substances (see table).

Microcrystalline Tests:

- 

Understand the reagent quality control system of the Controlled Substances Section. Identify various reagents used for crystalline tests as frequently used or infrequently used.

- 

Understand how to perform various microcrystalline tests and the expected results for commonly encountered substances (see table).

Testing Format

~.

The examination consists of short answer, matching, and multiple choice questions. Each question will have the point value indicated. There will be an opportunity to obtain extra credit during the exam. The trainee will be provided with a private, quiet area in which to take the exam; scratch paper and a calculator are permitted. The trainee will need to answer the questions correctly to pass and proceed with the training program. If the trainee does not pass the exam, he/she will be referred to the Laboratory Director.

Point Breakdown:

Total Points = 108 points Passing = 90% = 97 points Extra Credit = 6 points

Breakdown of points by topic:

Accreditation: 7 points

Drug Control Policies: 19 points + 2 bonus

Drug Classifications, Pharmacology, and Effects: 35 points + 3 bonus

Structures: 20 points

Spot Tests/ Screening Tests: 1-7 points

Microcrystalline: 10 points + 1 bonus

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MODUL~ O7

Controlled Substances Training Guide Version 2009

Subject: Midterm Written Exam

Page 3 of

Substance Control Effect / Use Spot Microcrystalline

Status Tests table

cocaine ~ stim cocaine A. A ~ Pt x

GHB 1 De ~eCl3 X

LS~ 1-A Hall Van Urk's X

Heroin ~ Narc SS Mar uis

x

Oxycodone 1 Narc SS Mar uis

X

MDMA 2 Hall Ferricyanide X

Mar uis

, Meth 1 St9m Ferricyanide Hanging Drop

X

. ~

nnar uis Ac Au, Pt

Hydrocodone 1 3 Narc S5 Mar uis

x

Amphetamine 2 Stim Mar uis X

Hydroxone 1 Narc SS hlar ~ X

Alprazolam 3 Benz De X

PCP 1 Hall Co . Au, KMnO4 X

Codeine 1 3 a Narc N r u X

Morphine 1 Narc N X

Barbituric acid 3 De an i X

Procaine n Urk's

Benzocaine Van Lrk's X

Toluene AVC Mar uis x

Pseudoephedrine cur r X

De estant

Carisoprodol D uscle X

Relaxant

Diazepam Benz De Janovsk X

Psilacine Hall Weber X

Fentanyl Narc S Mar uis

Dextro ro ax hene 3 Narc S X  
Guaifenesin Ex ec#orant Mar ~is x  
Dextromethor~ha~ Cough Marquis x  
Su resant  
~hen e~rioe Decon estant Mar uis  
Acetamion hen Anal eslc x  
Chfo heniramine Antihistamine  
Abbrev~atlons IJse~ in Table:  
Narc (N) — Natural Opiate Narcotic Stim — CNS Stirr~ulant  
Narc (SS) — Semisynthetic Narcotic Dep — CNS Depressant  
Narc (S) — Synthetic Narcotic Hall — Hallucinogen  
AVC — Abusable Volatile Chemfca! DD — Dangemus Drug  
~ffecti~e date 02-09-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY

Controlled Substances Training Guide MQDUL- a8  
Subject: Measurements and Sampling in Version 2-09  
Page 1 of 8

MEASUREMENTS AND SAMPLING READING LIST

(to be initialed when completed)

1) S. Bell, Forensic Chemist, 2006.

Ch. 2-"Statistics, Sampling, and Data Quality"

2) M. Coffin, et. al. "Representative Sampling of 'Street' Drug Exhibits," Journal of Forensic Sciences 38 (1993) pp. 641-648.

3) R.S. Frank, et. al. "Representative Sampling of Drug Seizures in Multiple Containers," Journal of Forensic Sciences 36 (1991) pp. 350-357.

4) D. Tzidonis and M. Ravreby, "A Statistical Approach to Sampling: A Case Study," Journal of Forensic Sciences 37 (1992) pp. 1541-1549.

5) C.G.G. Aitken, "Sampling - How Big a Sample of Forensic Sciences 44 (1999) pp. 750-760.

6) S.A. Coulson, et. al. "How Many Samples from a Drug Seizure Need to Be

Analyzed?," Journal of Forensic Science 46 (2001) pp. 1456-1461.

7) "Guidelines on Representative Sampling", ENFSI, 2004. [www.enfsi.org](http://www.enfsi.org)

8) SWGDRUG Recommended ed. "Part 111 A- Methods of

Analysis/Sampling Methods for Qualitative Analysis", February, 2006.

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Effective date 04-01-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 05  
Controlled Substances Training Guide Version 2009  
Subject: Measurements and Sampling Page 2 of 8  
OBJECTIVE

- To familiarize the trainee with the operation of laboratory balances.
- To familiarize the trainee with balance calibration and quality assurance.
- To familiarize the trainee with the recording and reporting of weights and volumes in laboratory notes.
- To familiarize the trainee with calculations for estimating the number of items in a submission based on the weight.
- \* To familiarize the trainee with calculations for determining number of average units in a submission.
- To familiarize the trainee with the concepts
  - To instruct the trainee on the sampling procedures in the laboratory.

DISCUSSION

- Measurements of Weight

In Texas the penalty for possession or delivery of a controlled substance depends upon the identification of a controlled substance as well as the aggregate weight of the controlled substance including salts and diluents. As a small difference in weight can result in a difference in penalty, great care must be used when determining the final weight to be reported. For example, the penalty for possession of a substance found to contain cocaine with an aggregate weight of 3.9999 g could currently result in a maximum 10 year sentence with a \$5000 fine. If the weight of the same substance is reported to be 4.0001 g, the maximum sentence becomes 20 years with a \$10,000 fine.

When reporting the results of an analysis and relying on these results, the analyst needs to have as much confidence in the weight reported as for the identification of an unknown substance. This requires that the balances used to determine weights be used properly, and that they be demonstrated to be working properly. It is also important that any calculations be clearly documented in the case notes and that conversion factors when used be clearly noted or referenced (as in the Training Guide or SOP).

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## HOUSTON POLICE DEPARTMENT CRIME LABORATORY

### Controlled Substances Training Guide MODULE 08

Subject: Measurements and Sampling Version 2009

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A discussion of weight determination should include defining the terms weight and mass

as an analyst may be asked to explain what they mean and what the difference is, if any, between the terms. Mass is an invariant measure of the quantity of matter in an object. The weight of an object is dependant upon the mass of the object and is a measure of the force with which gravity attracts the object. Because gravitational attraction is subject to slight geographical variation with altitude, the weight of an object is a somewhat variable quantity. For example, the weight of a beaker would be less in Denver than in Houston because the attractive force between it and the earth is less at the higher altitude. The mass of the beaker, on the other hand, remains constant regardless of the location in which it is measured.

Weight (W) and mass (m) are simply related to each other through the expression

$W = mg$  where g is the acceleration due to gravity

Scientific analyses are based on mass in order to free results from dependence on location. The mass of an object is ordinarily obtained by use of a balance that permits comparison of an object's mass with that of a known mass; because g affects both the known and unknown to the same amount, equality in weight also indicates an equality in mass. It is common to say, the difference between weight and mass is not always observed. The operation of comparing masses is called weighing and the objects of known mass as well as that of the process are called weights. As long as the comparison of known masses to known masses is performed at the same

location under the same conditions (balance), then the terms mass and weight may be used interchangeably.

The laboratory has a variety of balances available for use depending upon the level of accuracy required and the amount of material to be weighed. Top-loading balances typically have weights from a few kilograms to one-tenth of a gram and are the most frequently used. For weights requiring more accuracy, analytical balances are available with readability to 0.0001 grams but with a maximum load capacity of approximately 20 grams. For large items bulky or high-capacity balances are available which can tolerate loads of several hundred pounds.

The reported aggregate weight of a controlled substance should not include packaging unless specifically stated in the report. For example, "weight includes wrapping" for a cigar stub or cigarette stub or "gross weight" for sealed ampules of liquid which are retained and not opened. For cases which involve numerous containers, the analyst may choose to subtract an appropriate packaging weight from the gross weight instead of removing the contents of each and every container. Care must be taken when this is

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Subject: Measurements and Sampling Page 4 of

done so that the analyst does not use a packaging weight which is excessive or inefficient. All calculations must be documented in the case notes to support the final reported weight. If the final weight is at a cut-off, then the analyst may have no choice but to weigh the contents of containers separately.

When adding or subtracting weights the analyst should make sure that the same degree of accuracy is used for all weights. For example, a weight of 0.8 grams should not be added to a weight of 0.2034 grams for a combined weight of 1.0034 grams. Instead, the two weights should be reported separately or both weights should be determined on the same type of balance. In this example the sample weighing 0.8 grams might be 0.7545 grams or an analytical balance resulting in a combined weight of 0.9579 grams. Another concern when adding the weights of multiple samples is to ensure that the variability in the individual weights does not combine to produce a resulting total weight which is too high or too low. Consider the following examples where 3 samples have been weighed separately on a top-loading balance:

Sample A- 0.3 g ± 0.1 g Sample D- 0.2 g ± 0.1 g  
Sample B- 0.4 g ± 0.1 g Sample E- 0.3 g ± 0.1 g  
Sample C- 0.4 g ± 0.9 g Sample F- 0.4 g ± 0.1 g  
Total = 1.1 g ± ? Total = 0.9 g ± ?

The uncertainty associated with the sums could be as large as ± 0.3 g if the signs of the three individual variations happened by chance to be all positive or negative. Therefore, the weight of samples A+B+C could be anywhere between 0.8 to 1.4 grams and the weight of samples D+E+F could be anywhere between 0.6 to 1.2 grams. The best way to prevent the defendants in these cases from being charged with the wrong penalty range is to reweigh the samples on an analytical balance. The results could look something like this:

Sample A- 0.26 g ± 0.0001 g Sample D- 0.2413 g ± 0.0001 g  
Sample B- 0.4504 g ± 0.0001 g Sample E- 0.3401 g ± 0.0001 g  
Sample C- 0.3513 g ± 0.0001 g Sample F- 0.4432 g ± 0.0001 g

Total = 0.9640 g ± ? Total = 1.0243 g ± ?

Now the weight of samples A+B+C could be anywhere between 0.9637 to 0.9643 grams and the weight of samples D+E+F could be anywhere between 1.0243 to 1.0247 grams. There should no longer be a concern about the defendants being charged with the wrong penalty range.

When recording weights in the case notes, the analyst should document the reading exactly as displayed by the balance used. However, when reporting weights the following conventions should be used (see Reporting Guidelines in the CS SOP).

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## HOUSTON POLICE DEPARTMENT GRIM LABORATORY MANUAL

Controlled Substances Training Guide Version 2009

Subject: Measurements and Analysis of 8

Amounts between 0.01 g and 1004 g will be reported as such. Amounts over 1000 g will be reported in kilograms. Amounts less than 4.01 g will be reported as "Less than

0.01 grams". Amounts at a cut-off will still be reported to the first significant figure other than zero. For example:

15.2 g report as "15.2 grams"

1.0026 g report as "1.002 grams"

0.6 g report as "0.6 grams"

0.1703 g report as "0.17 grams" or "0.1 grams"

0.0341 g report as "0.03 grams"

0.0097 g report as "less than 0.01 grams"

1012.9 g report as "1.0 kilograms"

Note that weights are not rounded but are truncated (trailing digits are dropped off).

The weight ranges for controlled substances in Penal Code, or 4 in Texas are in metric units of grams. For marijuana the penalty is in ounces and pounds.

Unless, weights are measured directly in these units, conversion between metric and English units will be necessary when reporting results. The following conversion factors are to be used:

### Determination of

For Penalty Group 1 substances are not determined by weight but by abuse units. An abuse unit may be liquid form and is defined in the Texas Health and Safety Code as:

a single unit on or in any adulterant, dilutant, or similar carrier medium, including marked or perforated blotter paper, a tablet, gelatin wafer, sugar cube, or stamp, or other medium that contains any amount of a controlled substance listed in Penalty Group 1-A, if the unit is commonly used in abuse of that substance; or

each quarter-inch square section of paper, if the adulterant, dilutant, or carrier medium is paper not marked or perforated into individual abuse units; or

if the controlled substance is in liquid form, 40 micrograms of the

controlled substance including any adulterant or dilutant.

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Currently, the only substance listed in Penalty Group 1-A is lysergic acid diethylamide (LSD). Typically, LSD will be dissolved in a solvent which can be dispensed dropwise or applied to another medium such as paper or breath mints. Individual squares of paper or mirrors found to be laced with LSD would each be an abuse unit. If a sheet of paper with perforations or score lines on it is laced with LSD, then the number of abuse units is equal to the number of perforated squares even though they have not been separated. If the same sheet of paper does not have perforations, then the analysis must measure the paper and determine the number of quarter-inch square sections to which it equates. If a liquid is found to contain LSD, then the liquid must be weighed in grams and this value converted to abuse units by using the factor 40 micrograms = 1 abuse unit. ..

For example of liquid  
(20 drop number  
of abuse  
0.4774 g

Estimation

When dealing with large numbers such as tablets or capsules, the analyst may choose to determine approximate number of these items instead of actually counting them. This is easily determined if the total weight of the items is known and the weight of a sample number is known.

For example, a bottle of tablets is received and the weight of the tablets is found to be 256.4 grams. Ten of the tablets are found to weigh 8.0 grams. The approximate number of tablets in the bottle is determined as follows:

$$\frac{256.4 \text{ grams}}{8.0 \text{ grams}} \times 10 \text{ tablets} = 320 \text{ tablets}$$

Purity Determinations and Measurements of Volume

It is sometimes necessary for the purity of a controlled substance within a mixture to be determined. This is typically done for investigative purposes when a narcotics officer

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## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 5

### Controlled Substances Training Guide Version 2009

#### Subject: Measurements and Sampling Page 7 of 8

want to know if the dope he just purchased is any good. It is important for the analyst to be able to explain the difference between the identification of a substance

(qualitative analysis) and the purity of a substance (quantitative analysis) as the 1 she may be asked to perform a "quality" test when the person actually means purity or quantitative test.

Purity determinations are typically performed by dissolving a known amount of material into a known volume of solvent. The resulting solution will then be subjected to testing (UV spectrophotometry or gas chromatography) which measures the amount of the substance of interest without interference from other substances which may be present. This type of analysis would therefore require accurate measurement of both weight and volume. Volume is most often measured in units of milliliters or liters with the accuracy of the measurement dependent upon the container or device used (e.g., graduated cylinder, volumetric flask, etc.). A high degree of accuracy can be made moot with a low degree of accuracy in volume. The final accuracy required will determine the measuring devices to be used when performing such tests.

#### Sampling

Even before the weights of submitted items are determined, the analyst will perform a visual examination using the naked eye or stereoscope. This allows the analyst to separate evidence items into appropriate homogeneous groupings based on the appearance of the containers and contents.

Once such groupings have been made, the analyst must now consider how to sample the items for testing. The situation is when one item of evidence has been submitted and the analyst is faced with taking a representative portion (sample) to represent the whole. For example, if a bag of white powder is submitted, then a representative portion (sample) of that powder is tested fully and completely. It would not be prudent to consume all of the powder for testing as there would not be anything left for independent testing or viewing in court.

In certain situations the analyst has no choice but to use the entire evidence submitted to confirm the presence or absence of a particular substance. This can occur when only a small residue amount is received for testing. In those situations extra steps are taken to demonstrate that results obtained did not come from contamination of supplies or equipment used for testing.

Often the analyst is faced with taking representative samples not just from one item, but from multiple items. For example, a hundred ziplocks, each containing a white powder. Do all of the bags need to be tested, or can a representative subset be chosen to satisfy analytical and statutory requirements? The answer is determined by a laboratory's sampling plan. A review of the literature will give the analyst an idea of the various Effective date 04-1-05

## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MANUAL ~ 8

### Controlled Substances Training Guide Version 2009

#### Subject: Measurements and Sampling Page 8 of 8

sampling plans that have been used by laboratories over the years. They may range

from taking samples from the square root of the total items received to simply testing each and every item received. Some of the plans are non-statistically based such as the square root method. Other sampling plans are statistically based and allow the analyst to make an inference about the whole population from the results of the samples tested. An analyst must be familiar with his/her laboratory's sampling plan and how to apply it properly to various case work situations. See the Controlled Substances Section SOP for the current sampling plan used.

#### PRACTICAL EXERCISES

- 

The trainee will receive a practice worksheet with exercises to be performed related to weights, volumes, and associated calculations.

- 

The Trainer will demonstrate how to perform calibration checks on the various balances available in the laboratory.

+ Once the trainee has completed the practice it will be reviewed with the Trainer.

#### DOCUMENTATION ~~~

The Trainer will review the proper E-File documentation for noting weights,

volumes, balances used, and any ~ performed. The Trainer will review the

proper documentation for calibration checks of the balances available

for use in the Controlled Substances

#### STUDY GOALS ~

- 

Define the terms assay, weight, qualitative analysis, quantitative analysis, accuracy, precision, homogeneous, and heterogeneous.

- 

Understand the quality control procedures for the various balances used by the Controlled Substances Section. Be able to demonstrate calibration and calibration checks for the same.

- 

Be able to convert between metric and English weights.

-

Be able to calculate the number of abuse units for Penalty Group 1-A controlled substances.

Be able to approximate the number of items given the total weight and an item

weight,

Understand the sampling plan used by the Controlled Substances Section.

Effective dates

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Sub' t: UV 1 VI ectrn hotome pa e 1 of 11

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IVIS SPECTROPHOTOMETRY READING LIST  
(to be initiafed when completed)

1) CND Anatyical: Series of Analytical Pro~fes  
Forensic and Analytical Chemistry of Clandestine Phenethylamines  
Ch. 4 — "Ultraviolet Spectrophotome#ry"

2} A.C. Moffat editor, Clarke's Anal sis of Dru s and Poisons, 3~ Edition, 2004.  
Ch. 2~ — "Ultravivfet, Visible, and Fluorescer~ce Spectropho#ometry"

3) 5. Gorog, Ultraviolet-Visibfe S ectro hotomet in F'ha aceut~caE  
Ana#ysis, 1995.  
Ct~. 1 — "Introduction"  
Ch. 2—"The Measurement of Light Abso ti flectior~}"  
Ch. 3—"Qualitati~e Analysis: Relatio e the Str~ctur~ and

Spectra of Pharmaceutical Co s

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## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MANUAL

Controlled Substances Training Guide Version 2009

Subject: UV VIS Spectrophotometry Page 2 of 1

### OBJECTIVE

- To familiarize the trainee with the theory and application of UV-VIS spectrophotometry in drug analysis.
  - To familiarize the trainee with the UV-VIS spectrophotometry instrumentation and software used in the laboratory.
  - To familiarize the trainee with the quality assurance procedures for the UV-VIS spectrophotometer.
  - To make the trainee aware of the advantages, disadvantages, and limitations of UV-VIS spectrophotometry.
  - To familiarize the trainee with the application of UV-VIS spectrophotometry in the quantitation of substances of interest.
- ### DISCUSSION
- Introduction to Electromagnetic

Gamma x-ray IS IR Microwave Radio

High Energy Low Energy

Translations Nuclear Inner electron Vibrations Rotations

Wavelength 10<sup>1</sup> to 10<sup>13</sup>

{meters}

Frequency 10<sup>13</sup> to 10<sup>5</sup>

(waves 1 sec)

Wavenumber 10<sup>5</sup> to 10<sup>13</sup>

(waves 1 cm)

The electromagnetic spectrum is a continuous range of radiation. The wavelength or frequency of the radiation defines the position of the electromagnetic spectrum. The shorter the wavelength, the higher the frequency, and the greater the energy of the radiation. The relationship between wavelength ( $\lambda$ ) and frequency ( $\nu$ ) is described by the equation

$C = \lambda \cdot \nu$  where (C) is the velocity of radiation in a vacuum

This inverse relationship explains why shorter wavelength means greater frequency. To overcome this confusion the term wavenumber is sometimes used and is equal to  $1/\lambda$  with units of cm<sup>-1</sup>.

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Sub~ect: UV 1 VIS S ectro hotomet Pa e 3 of 11

Soectroscopy is the study ~f the interaction of electromagnetic radiation. with\_ matter. This interaction of radiation witF~ matter can cause redirection of the radiation andlor transitions between the energy leve~s vf the atoms within molecUles. A transition from a lower level to a higher level with transfer of energy from the radiation field to the atom ar molecule is called absorption. A transition from a higher le~el to a lower le~el is called emission (fluorescer~ce). Redirection of radiation is cailed scatter and may or may not occur with the transfer of energy.

When atoms or molecu~s absorb radiation~, the incoming energy excites a quantized structUre to a higher energy level. The type of excitation depends on tf~e wavelength vf the raciation. Electrons in their outer orbital {~alence electrons} are promoted to higher orbita~s by UV vr Visible radiation. The absarption of infrared radiativn cac~sss ~ibrational excitation of malecules and low energy infrared micrawave radiation results in rota#ional excitation of molecules. It requires ion of very s~ort wavelengths in the X-ray region to excite inner orbital atomic el tro to a higher state. Radiativn of even shorter wave~engths makes up #he ray region of the electromagnetic sp~ctrum. It is transitions in atomic e # suit in the emission of this hig~energy, penetrating radiatian. ~

~~

. Theory of UVNIS Absorprfon

U#tr~violetNisible spectrophotnme#ry ~violet and visible el~ctromagnetic radiation (or light) ta excite a compot a e(o~ter s~ell) electrons into a higher energy state. The UVNIS spectrum ~s from 2~0-80Q nanometers, but it is the UV portion fram --200~00 nm whic~ useful in general drug ana~ysis.

When the light is passed thro a~ple, ~alence electrons from the lower, occupied molecular orbitals (groun t s re excited to the next higher, unoccupied orbitals (excited states). In e ou s ate, ~alence electrons of three typss exist in bondir~g and non-bonding orbi

1. 6— ele

s e the electrons formfng single bonds such as the GC and GH bonds. he a orbitals are characterized by their low energy level. T#~ey are strongly held and are th~refore difficult to excite.

2. n— electrons form multip~e bonds including C=C and C=O. The energy levels of n orbitals are higher. The rc electrons are more looseiy held and are therefore easier to excite.

3. n— electrons are non-bonding unshared electron pairs of hetera atoms in organic compounds and i~clude the lone pair af electrons on ~itrogen and the two lone pafrs on oxygen.

The high energy excited states of v and n bonds are refeRed to as antibonding orbitals and are designated v\* and n\*. When ultra~iolet iight in the range of 2Q0-400 nm is absorbed, it is typicalEy double bond n electrons which are excited from a n orbital

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HOUSTON P01.fCE DEPARI'(IAENT Cr'ciiv9~ Li+~si~RA i vriY MODUL~ d9.1

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Sub'ect: UV 1 VIS S ctro hotome Pa e 4 of 11

ground state,  $E^\circ$ , to  $n^*$  orbital excited state,  $E'$ . The amount of energy required for this transition can be related to frequency as follows:

$$E \sim n^*$$

$$E' - E^\circ = E$$

$$E^\circ \sim$$

$$E = h\nu \text{ where } h \text{ is Planck's constant (units of Jsec)}$$

### Instruments

Spectrophotometers are the devices used to separate the numerous wavelengths within a range of radiation and allow for the absorption of the wavelengths by compounds of interest to be measured independently. The typical output from such an instrument is an absorption spectrum and its plot of radiation as a function of wavelength or energy. As only certain transitions from a lower energy state to a higher energy state are allowed for atoms, an absorption spectrum can be a useful tool for the identification of the species.

A UV-Vis spectrophotometer usually has the following components including a radiation source, a wavelength selector, a sample, a detector, and a readout device.

1. Source — creates energy in the desired region

Transmits visible light

Directs UV light

2.

Monochromator selects desired band of radiant energy via a diffraction grating or (older and cheaper instruments).

3.

Sample Cell — vessel for holding sample (cuvette). Quartz cuvettes are used because plastic or glass cells will absorb the UV light.

4.

Detector — device for measuring unabsorbed (transmitted) radiant energy having passed through the sample. Photomultiplier tube (or diode array) used to convert incident light into electrical energy. Includes an amplifier to increase the signal from the detector.

5. Recorder — produces spectrum (graph of absorbance vs. wavelength).

Two types of spectrophotometers are available and are referred to as single-beam and double-beam. Single-beam spectrophotometers are primarily used to monitor absorptions at a single wavelength although they can be used to generate full spectra over a range of wavelengths. A measurement is taken with a reference cell to serve as a background. The reference cell is removed and replaced with a sample cell which is then scanned over the same range. The reference scan is ratioed with the sample scan to produce the final absorbance spectrum.

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## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE #9.1

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Subject: UV / VIS Spectrophotometry of 5 of 11

In a double-beam spectrophotometer, the radiation from the monochromator is split into

two identical beams by a beam splitter (chopper or rotating mirror). One beam passes through the sample cell and the other through the reference cell, before being focused onto the detector alternately. See Section 3.3.1 of the Shimadzu Manufacturer's Instruction Manual (book #1) for a schematic of the UV spectrophotometer currently used by the Controlled Substances Section.

### Interpretation of Spectra

While the wavelength of light absorbed is determined by the difference between the energy levels of the ground and excited states of the electron, the amount of light absorbed by a substance is proportional to the number of molecules in the path of the light. A spectrophotometer determines the amount of light absorbed by measuring the intensity of light at each wavelength before and after exposure to the substance of interest in solution. These values are related to a term known as transmittance,  $T$ , where

$T = \{I / I_0\}$   $I_0$  is the incident radiation

$I$  is the transmitted radiation

Older literature would often plot UV spectra as a function of transmittance vs. wavelength or percent transmittance (%  $T = I / I_0 \times 100$ ) vs. wavelength. Unfortunately, transmittance is a logarithmic term and is not linear. The more common convention is to plot UV spectra as absorbance ( $A$ ) vs. wavelength where

$$A = \log_{10} (I_0 / I)$$

This absorbed electromagnetic radiation ( $A$ ) is directly or linearly proportional to the concentration ( $c$ ) of the sample as well as the path length of the radiation ( $b$ ) and a term known as absorbance coefficient of the species. This relationship is known as the Beer-Lambert Law and is expressed mathematically as

$$A = (a)(b)(c)$$

This equation will be discussed in greater detail under the topic of UV quantitation. UV spectra generated over the range of 220-340 nm are useful for detecting substances that contain unsaturated functional groups such as  $C=C$ ,  $C=O$  and substituted aromatic rings. The term chromophore is used to identify a functional group which absorbs UV/VIS light. If two substances possess the same chromophore and their UV spectra are collected under the same conditions, then the spectra will look the same. This means that UV spectrophotometry should be utilized as an instrumental presumptive test and not as a confirmatory test as the UV spectra are not unique. Effective date 04-09-08



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Subject: UV / VIS Spectrophotometry of 11

In addition to whether a substance has a chromophore or not, several other factors can affect the appearance of a spectrum. First, UV spectra are not generally sharp bands over a narrow wavelength range. Instead they appear as broad absorptions sometimes over several wavelengths. This is partially due to the presence of vibrational and rotational levels in a molecule's electronic states. When a molecule absorbs UV light it can be excited from the ground electronic state into these levels of the excited state resulting in band broadening. The peak of these bands is identified by its wavelength as  $\lambda_{max}$ .

Another factor which plays a significant role in the appearance of a UV spectrum is the solvent used to prepare a sample solution. Changes in polarity and pH of solvents (methanol, ethanol, aqueous acid, or aqueous base) can change band shape, the location of peak maxima as well as the absorption intensity. The shifts of absorption bands can be useful in determining what type of substance is present in a sample and are termed as follows:

~~

1.

Bathochromic (red) shift = shifting of  $\lambda_{max}$  wavelengths

2.

Hypsochromic (blue) shift = shifting shorter wavelengths

3.

Hyperchromic shift = increase in intensity of a band

4. Hypochromic shift = decrease in intensity of a band

All of these terms refer to relative change in value under a given set of conditions to another value under a different set of conditions. Examples include:

7.

When morphine is dissolved in acid, it produces a UV peak with a max at 284 nm. When it is made basic, the peak broadens and demonstrates a bathochromic (red) shift to 294 nm.

2.

When amoxicillin is in a strong base (pH 13), it produces a UV peak at 254 nm. When the pH of this solution is decreased to -- with HCl, the peak demonstrates a hypsochromic (blue) shift to 235 nm.

3.

The pH adjustment of barbiturate solutions which produces a blue shift in wavelength will typically increase the intensity of absorption resulting in a hyperchromic shift.

4.

The most straightforward example of hypochromic shifting is to dilute a solution of an absorbing substance thereby decreasing its concentration and observing the corresponding decrease in intensity of absorption.

The UV spectrum of benzene shows two absorption bands one at 204 nm and the other

at 254 nm. Only the second weaker band would be seen in the scan range of 220-340 nm. In this case benzene itself is a chromophore as it is the conjugation of the entire ring which results in these absorption bands.

In amphetamine a substitution is made at one of the carbons of benzene. This results in a monosubstituted benzene compound. The side-chain of amphetamine does not  
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result in a new absorption band (it is not a chromophore), but it does decrease the symmetry of benzene and has an effect on the band shape at 254 nm causing a small bathochromic shift to 257 nm and a characteristic "3-finger" shaped band. Functional groups which influence other chromophoric groups but do not possess or have very slight UV activity of their own are called auxochromes. In the case of amphetamine and other phenethylamines such as methamphetamine, ephedrine, etc., the chromophore is considered to be the monosubstituted benzene ring with the side chain acting as an auxochrome.

Another example of an auxochrome effecting the absorption spectrum of a compound is the methylenedioxy group of the designer drugs MDA, MDMA, etc. This group has two electron rich oxygen hetero atoms in conjugation with the benzene ring and is an example of a disubstituted benzene. This auxochrome produces bathochromic shift of benzene's two bands from 204 and 254 nm to 230 and 280 nm respectively. The "3-finger" shape of the lower energy band is lost and an associated hypsochromic increase in intensity is observed.

The best way to identify the source of a UV spectrum (stance may be present) is to compare it with spectra recorded under identical conditions including instrumental methods, solvent, wavelength range, and appropriate concentration. The analyst must remember that these results are presumptive and further testing must be performed to confirm the presence of indicated substance. It is also important to remember that the absence of a UV spectrum does not mean that there is "nothing" present in a sample. It simply means that the substances present do not absorb or cannot be detected under those conditions. Examples include carisoprodol which has no absorption between 220-340 nm and barbiturates run in aqueous acid.

#### QUANTITATION BY SPECTROPHOTOMETRY

In addition to being a tool for indicating the presence of a substance in an unknown sample, Vis spectrophotometry can be used to quantitate or determine the amount of that substance present in the unknown sample. This is based on the Beer-Lambert Law mentioned earlier but discussed in more detail now. As absorbance (A) is a unitless term, the factors (a), (b), and (c) must combine to cancel their units. The pathlength (b) is generally accepted to be 1 cm so choosing a concentration (c) will determine the units of {a}. In the Controlled Substances Section concentration is measured in mg/ml so absorptivity (a) would have units of ml / mg cm.

$A = \{a\}(b)(c)$  where A = Absorbance  
a = absorptivity in ml / mg cm  
b = path length in cm  
c = concentration in mg / ml

The absorptivity is a constant for a particular substance at a particular wavelength. Each wavelength in an absorption spectrum for a substance has an associated Effective date 04-09-09

absorptivity \_ e~en \_ if it is \_ zero. The actual value for a sample must be determined experimentally, although references such as Clarke's will often provide this information. Analysts must remember that the value for absorptivity is concentration dependent so it may differ from one source to the next. For example the value for absorptivity experimentally determined for the 233 nm peak of cocaine in the Controlled Substances Section is 42 ml / mg cm. In Clarke's 3rd Edition it is cited as 430 with units of 100ml / g cm because the values in Clarke are determined from 1% solutions.

#### • DETERMINATION OF E VALUES

To experimentally determine the absorptivity for a particular substance at a particular wavelength (generally taken to be a peak maxima), the analyst would simply prepare a known concentration solution for that substance, place it in a single cuvette of known pathlength (width), and measure the absorbance at the wavelength and the absorbance at a background wavelength.

In the Controlled Substances Section the equation is simplified by always using sample cuvettes of 1 cm pathlength. This in effect term to "drop out" of the equation. The terms absorptivity and pathlength  $l$  to produce a new term called the E value which is only concentration dependent.

$A = (a)(b)(c) = (E)(c)$  this can be solved for E as such  
E value =  $A / L$

weight in mg

For example to determine the E value for cocaine at 274 nm, 9.0 mg of standard cocaine base is dissolved in 25 mL of 0.1 N HCl. This solution is placed in a 1 cm quartz cuvette and the absorbance at 274 nm is measured to have an absorbance of 1.23. A background absorbance at wavelength 300 nm is measured to be 0.035. The E value is calculated as follows:

$$E \text{ value} = (1.23 - 0.035) / (25 \text{ mL}) = 3.4$$

(9.0 mg)

#### DETERMINATION OF PURITY

Once the E value is known for a substance it can be used to determine the purity of that substance in an unknown sample. The above equation can be rearranged to solve for the weight of the substance of interest in mg:

$$\text{Substance weight in mg} = (A_{\text{sample}} - A_{\text{background}}) / (E \text{ value} \times \text{Vol solvent in mL})$$

(E value)

This value can now be used to find purity in an unknown sample as follows:

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% Purity = Substance weight in mg x 100

Sample weight in mg

Combining the two equations into one yields

% Purity =  $\frac{(A_{\text{sample}} - A_{\text{blank}}) \times 100}{E \times d \times \text{Conc}}$

(E value)(Sample weight in mg)

For example to determine the purity of cocaine base in a white powder, the analyst weighs 14.2 mg of the powder and dissolves it in 25 ml of 2/3 N H<sub>2</sub>SO<sub>4</sub>. This solution is placed in a 1 cm quartz cuvette and the wavelength at 274 nm measured to have an absorbance of 0.926. A background absorbance at wavelength nm is measured to be 0.090. The E value was determined above to be 3.4 at this length. The % purity is calculated as follows:

$$\% \text{ Purity} = \frac{(0.926 - 0.090) \times 100}{(3.4)(14.2 \text{ mg})} = 52 \%$$

### CONVERSION FROM BASE

All E values in the Controlled Substances Manual are given for the free base form of substances. This means that if a sample of a substance is determined the purity can be converted to that form by using molecular weight of the free base form and of the salt form as follows:

% Purity of base = % Purity of salt x  $\frac{\text{MW base}}{\text{MW salt}}$

(MW base)

For the cocaine example above, an FTIR spectrum determines that the cocaine HCl form is present. The % purity of cocaine as the salt form is calculated as follows:

$$\% \text{ Purity of Cocaine HCl} = 52\% \times \frac{303}{309} = 50.4\%$$

### PRACTICAL EXERCISES

o

The trainee will receive a list of practice samples for performing UVNIR analysis including quantitation.

The Trainer will demonstrate how to perform qualitative UVNIR analysis using acids and basic solvents as well as how to perform pH and UV shifts.

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- Once t~e\_trainee~\_has\_comQleted ths q~~!itati.~e por:i~~ of tha pratitice ~~rks~aet it will be re~iewed with the Trainer.

- The T-ainer wilf demonstrate how to prepare sampfes for quantiation as well as how to perForm #he necessary calculations for purity and E-value determinations.

\*

Once the trainee has completed t~e quantita#ive portion of the prac#ice worksheet it will be reviewed with the Trainer.

#### D~CUMENTATION

The Trainer will review the proper labeling of UVN15 spectr including quantitation {purity} calculations. ~

#### STUDY GOALS

- Understand the principles of electron excitati energy absorption and which types of excitations are respar~sible for ro' observed spectra.

- Define the terms "chromophore", "hypso ic hift", "bathochromic shift", "hyperchromic shift", and "hypochro n. `

- Understand why spectra for nces may look simiEar ar~d why some substar~ces do no# produce

- Know the principle com both single beam and double beam instrument:

- Know the ger~eral~ nge ar~d units of ineasure as well as the acceptable absorbance r~pe a l UVNIS spectrum.

- Be able to tch vided UVNIS spectra ta a list o€ possible substances inclt~ding t~e ing:

Acetaminophen

Alprazolam

Amphetamines (Amph, Meth, Ephed}

Benzocaine and Procaine

Barbiturates (Amo, Seco, Pheno, etc.)

Caffeine

Carisoprodof

Cocaine

Codeine

Designers (MDA, MDMA, MDE)

Diazepam

Heroin

:ffec~f~e aate aa-os-os

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Ketamine

Lidocaine

LSD

Methadone

Phencyclidine

Know the equation to relate Absorbance to Transmittance and the Beer-Lambert Law

$$A = \log (1/T) = abc$$

where A is absorbance and T is Transmittance

a is a constant specific to the substance of interest at a particular wavelength

b is the pathlength in centimeters

c is the concentration in mg / ml (in the Controlled Substances Section)

Be able to calculate % purity of a sample from a provided VIS spectrum.

~

Be able to UVNIR spectrum.

. Be able to free base forms of

provided

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Contro~led Substances: Training Guide

Version 20Q9

S~b'ect: Ultra~ioleWisible S o hotomet hirnadzu Pa e 1 of 4  
SHIMADZU

-----~  
ULTRAVIOLETNISIBLE SPECTROPHQTOMETRY ~UVNIS)  
Instrument  
Shimadzu UV-2401 PC UVNIS Spectrophotometer  
Software

UVProbe

Instrument Startup:  
Turn on the instrumer~t, the computer {if necessary), ~r, and the printer.  
Allow the instrument to warm-up for ~15 minutes before ~ding.

2.  
if a window appears that requests a password ~ Do not enter any information.
3.  
Open the UVProbe software from the desktop
4.  
Ensure there are no c~ttes in the ins#n~ n, click on the Connect button near the bottom of the control ~an~ b in the instrument initialization sequence. ~  
~. When the initialization is complete with results on the screen and note any parameters which Fail (red marks). If any parameters Pass (green marks appear), then click OK and the initialization is in the logbook. If any of the parameters have failed, consult the Troubleshooting Section 4.2 of the Manufacturer's Instruction Book (book %Z) for remedial action. If corrective measures do not correct the problems, then consult with the assigned analyst(s) or Lab Manager and instrument off-line until the issue can be resolved.

fi.  
After successful initialization click the Baseline button on the control panel to set the baseline instrument. Make sure there are no c~ttes in the instrument parameters are set to start at 340nm and to end at 220nm,

then click OK the baseline runs along with the time in the logbook.

7. The instrument is now ready to acquire data.  
Instrument Shutdown:



~. Close the UVProbe software by clicking the X box at the far top right of the screen.

2.

A window will appear stating there is unsaved information; click the Yes button.

3.

Turn the instrument, the computer monitor, and the printer off. Turning the computer off is optional.

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Sub'eck: Ul#ravfvleWisible S ectro hotome# hirr~adzu Pa e 2 of 4

Data Acqulsttion:

1.  
Prepare the sampfe c~vette and place it i~ the sample cell.
  2.  
Fill another cuvette with neat preparation solvent and place it in the reference ce11.
  3.  
Click the Start button.
  - 4• When the New Data Set window appears you may enter your initials in the artalyst freld and the lab number in the comments field, then click OFC. If you click Cancel the data will be deleted.
  5.  
Cfick the Peak Pick button (four~h butto~ from the righ# on the toorbar).
  6.  
Right click in the peak pick area and uncheck Show vallevs. This will remove ths absorbance Minima data from the chart.
  - T.  
If all the peaks of interest are r~ot labeled then right click the peak freld and click properties. Enter the number 1, 2, oc 3 in the point and hit enter.
  8.  
To o~tain a background absorbance click on the Go To L on on the lower control panel and enter the desired wavelength, t er. The absorbance value for the wavelength is displayed on th TE: Tf~~s function will work only if your sampEe is stili in the inst
  8.  
If this is the first anafysis of the day clic~t th ., then properties. Click the Disabled bvtton for "Store All Data ~ Sin ile", then click Close.
  9. Click the print butto~ to print the re ort
- Changing Wavelength Range: ,~~
1.  
To scan across a different ~th region (e.g. 300nm to ~90nm for GBL analysis) click the "M" bu n( venth button from the righ# on the tooibar) and enter the desired start avelengths in the appropriate ~elds. Then clic~C OK. Do not change th r values.
  2.  
Perform a ba eli he new wavelength values before proceeding with the analysis.
  3.  
When yo re fr with your analysis, rese# the parameters to start a# 340r~m a~d end a On ia the "M" button.
  4.  
Pe~form a ba e scan on the standard wavelength region.

5. Note the baseline scan and method modifications in the logbook.  
instrument Performance Check (performed quarterly or as needed):

1.

Follow the routine Startup procedures before performing the two part Instrument Performance Check.

2.

First check the wavelength accuracy by using the two characteristic wavelength peaks of deuterium light at 486.0 nm and 656.1 nm. See the Periodic Maintenance Section 4. of the Manufacturers Instruction Manual (book 1) to reference the following procedure:

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A. Click the "M" button on the toolbar to modify the Method Parameters as follows:  
Select the Instrument Parameter tab

- a. Change the measuring mode from Absorbance to Energy
- b. Leave slit width at 0.2 nm
- c. Change source lamp from off to D2
- d. Change PM gain from Q min to 2 min

2. Select the Measurement tab

- a. Change wavelength from 340 — 220 nm to 650 — 650 nm
- b. Change scan speed from fast to medium
- c. Change sampling interval from 0.5 to a checkbox
- d. Click OK

B. Ensure that the cuvet holders are empty and click button.

C. When the scan is complete, enter the analysis name and the following info in

the comment box:

Wavelength Accuracy Check

Pass Range = 655.8 — 656.0 nm

Click OK.

D. Adjust the plot so that the 656 nm is on scale.

E. Right click in the Peak Pick and uncheck Show Values.

F. Right click in the Peak Pick box and select properties. Note the current threshold setting (usually 1) and adjust the threshold so that only the 656 peak is identified (usually 1).

G. Click Print

H. Follow the procedure for the wavelength range 490 — 480 nm.

I. When the scan is

complete, enter the analysis name and enter the following info in comment box:

Wavelength Accuracy Check

Pass Range = 485.7 — 486.3 nm

Click OK.

J. Adjust the plot so that the 486 nm peak is on scale.

K. Right click in the Peak Pick box and select properties. Adjust the threshold so that only the 486 peak is identified (usually 3-5).

L. Click Print.

M. Click the "M" button on the toolbar and return the Method parameters to normal values (see A.1. and A. 2.)

N. Re-run the baseline:

O. Right click in the Peak Pick box and adjust the threshold to the original value (see F.)

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3.

Once the wavelength accuracy has been checked, the absorbance accuracy of the instrument is verified. To do this weigh three samples of one of the validated standards (currently methamphetamine, heroin, or cocaine) and perform a quantitation using the experimentally determined E value. The determined purity should be within 10% of the expected value.

4.

If the instrument fails any part of the performance check, then consult with the assigned analyst(s) or the Lab Manager and take the instrument off-line until the issue can be resolved. If necessary, contact the Shimadzu service representative to perform necessary maintenance.

5.

Record the Instrument Performance Check results in the logbook as well as any maintenance performed. Store the printouts in the appropriate location.  
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Subject: Separations and Extractions Page 1 of 1

----- SEPARATIONS  
AND EXTRACTIONS READING LIST  
To be initialed when completed?

1) CND Analytical: Series of Analytical Profiles  
Forensic and Analytical Chemistry of Clandestine Phenethylamines  
Ch. 2 — "Extraction and Purification"

2) S. Bell, Forensic Chemistry, 2006.

Ch. 4- "Sample Preparation...", pp. 85-107  
Effective date 4-20-09

## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 1

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Subject: Separations and Extractions Page 2 of

### OBJECTIVE

- To familiarize the trainee with the fundamentals of separation theory.
- To familiarize the trainee with extraction techniques for separating components of a mixture.

### DISCUSSION

#### • Introduction

In forensic drug chemistry, the main purpose of an analysis is to identify the presence of controlled substances or dangerous drugs in submitted samples. This task can be made more difficult by the fact that almost all drugs interact with other substances such as sugars or starches or possibly drugs. While some tests are sensitive to the presence of interfering substances IR results are based on

all absorbing species present), other tests are sensitive to interferences

because they don't respond to these substances do not absorb in the UV

region). Some of the most useful techniques are able to separate the components of a mixture as well as mixtures (TLC and GC/MS). Because of the wide variety of substances encountered in drug analysis, the analyst needs to know the limitations and advantages of the testing procedures, as the selection of the right tests can greatly reduce the time involved in making identifications. Before certain tests can be conducted, the analyst may be required to perform separations to isolate or remove sample components. There are different ways to achieve this and one of the simplest takes advantage of varying solubility properties of chemical compounds. Solubility is the ability of two or more substances to form, without a chemical reaction, a homogeneous dispersion. More specifically, the solubility of a solid in a liquid refers to the concentration that is reached when a fixed amount of liquid has dissolved all of the solid it can hold at equilibrium (at a given temperature) to produce a saturated solution. While solubility is a physical property for a pure substance in a pure solvent at a specific temperature, the relative solubility of solids in liquids ranges from very low to very high values and can be conveyed by use of terms very soluble, soluble, moderately soluble, slightly soluble, and insoluble. Strictly speaking, no substance is absolutely insoluble, although for all practical purposes many substances appear to be so.

The most common solutions in general chemistry are aqueous based where water is the solvent. Water and other polar solvents such as methanol and ethanol are able to

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S b~ect: Se aration~s and ~xtractions Pa 3 of 9

dissolv\_e polar \_soE~#es based. on \_the ~rinciple . of ."like diGsol~ses like". ~!~f~ny drugs ~f

interest are polar within certain pH ranges so that the pH of a sof~tion can affect the solubility of that drug in water. Some substances do not have a polar character and are not very so~uble in water, but are freely solub~e in nonpo~ar organic sofvents such as chloroform or dichloromethar~e. All saft farms are considered polar and ther~fore dissolve in polar sol~ents. As a general rule, all free acids and free bases are ~onpolar and therefore will dissolve in most organic sofvents. Those compounds that will not

frm salts are neutral and usually prefer organic solvents. For example, alkaiine compounds like cocaine base form safts with acids and are therefore soluble in aqueous acidic solutions, but are insoluble in basic solutions. Likewise, acidic compounds like the barbiturates form safts with a(katine solutions and are t refo~e soEuble in basic aqu~ous solutions.

Determining the exact solubility of a su~stance is no ~'!!' #or drug analysis; however, knowing the approximate so~ubility pro s bstances is extremefy useful for the separation o# consti#uents in simple e mixtures. Texts such as Clarke's list relative solubilities in various so#~en n be useful references when trying to determine the best sol~er~t for a parti

Miscibility is a term used for iiquids to "c e extent of solubility of o~e liquid in another. Some liquids wi~l mix with r i !! proportions and are said to t~e miscible wi#h water. Such liquids are usual ubstances with polar character similar to that of water (the alcohols). The n ar uids like chloroform or t~exane do not have an effective attraction with pola wat r olecules so they are effective "squeezed out". S~ch liquids are said to ' cible with water. Other liquids, such as ether are sligt~tly soluble in w~ d said to be partially miscible. Two layers are formed when two immis~ibfe li re ir~ contact with each other.

Extraction procedure3`~~ased on relative solubility of a compound {solute} between two immisci~le solvents is a wide~y used method of drug isolation. One of the sofvents is ust~ally water and the other is a nonpolar organic such as cnloroform. Extraction involves bringing the two solvents ir~to intimate contact by exte~sive mixing, allowing the mofecufes of interest to partition from one sofvent to the other. The comp~~teness of t~e partitioning process will be determined by the compouncfs solubility in each sol~ent as well as t~e volumes of each solvent.

. Direct Solvent "Dry" Extractfon

This technique is based on the premise that the substance of interest in a sample mixture is soluble in a specific solvent while afl other components of the sample are

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acid #or acidic drugs).

2.

Acid and neutral drugs can be extracted from aqueous acid medium with an immiscible organic solvent.

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3.

Basic and neutral drugs can be extracted from aqueous basic medium with an immiscible organic solvent.

4.

Morphine is a special case and must be extracted from a pH 5 solution to a mixture of butanol and chloroform.

•

#### General Extraction Procedure

Using the principles above, a general extraction procedure can be devised which would be able to isolate strong acids, weak acids, neutrals and bases from a mixture which contains each of these substances:

Start) Dissolve mixture in 2/3 N H<sub>2</sub>SO<sub>4</sub>

Step 1) Extract the 2/3 N H<sub>2</sub>SO<sub>4</sub> with CHCl<sub>3</sub> (save the acid solution for step 2)

Step 1A) Extract the CHCl<sub>3</sub> from step 1 with 5% NaHCO<sub>3</sub> (this will contain the

strong acid drug)

Step 1B) Now extract the CHCl<sub>3</sub> from step 1A with H<sub>2</sub>O (this will contain

the weak acid drug)

Step 1C) Evaporate the remaining CHCl<sub>3</sub> to dry over the neutral drug)

Step 2) Add base to the saved acid solution until the pH ~ 10

Step 2A) Extract the basic solution from step 2 with CHCl<sub>3</sub>

Step 2B) Extract the CHCl<sub>3</sub> from step 2 in 2/3 N H<sub>2</sub>SO<sub>4</sub> (this will contain the

basic drug)

These same steps are presented as outlined below showing the final destination of each component from a mixture of ibuprofen, carisoprodol, and cocaine:

Start) Dissolve substance in 2/3 N H<sub>2</sub>SO<sub>4</sub>

Step 1) for general basic drugs

then

extract with CHCl<sub>3</sub> add sat Na<sub>2</sub>CO<sub>3</sub>

Step 1A)

extract with extract with evaporate extract

5% NaHCO<sub>3</sub> 0.45 N NaOH to dryness with CHCl<sub>3</sub>

Step 1B)

strong acid weak acid neutral extract with

ex. aspirin ex. barb ex. carisoprodol 2/3 N H<sub>2</sub>SO<sub>4</sub>

1  
basic dnag  
ex. cocaine

ff8~trv~ asce oa-za-as~

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Sub'ect: Se aration and ~xtractions Pa e B of 9

This general extraction\_proc~dure\_\_can\_be\_modified to f~c~!s on ~impl~r and r~ora li~eiY  
mixtu~es as derrtonstrated ~n the following examples.

• Removal of Asplrjn from a Mixture:

Aspirin will extract into chlorofarm easily from acidic solutions. Because it is usually present in ~arge quantities when mixed with basic drugs, several extractions are necessary to remo~e it all. Aspirin can be extracted frvm chloroform by washing with 5% sodium bicarbonate.

1.

Separation of weak acid (barbiturate) from aspirin:

Step 1} Dissolve sample in 2/3 N HZS44

Step 2) Extract 2/3 N H2S0¢ with CHC~~

Step 3) Wash the CHCl3 from step (2) 3 wit % NaHCO3 (the aspirin will go into the 5% NaH

Step 4} Extract tl~e CHCl3 from ~#,g~3 #~ .45 N NaOH to reco~er the barb ~~~

2.

Separation of basic drug ~ex. coc~r~ or~deine) from aspirin:

Step ~} Dissolve samp ~l H 504 and make the solution basic with sat Na2

Step 2) Extract tha t 03 solution with CHCl3

Step 3) Wash 13 from step (2) 3x with 5% NaHCO3 (the

aspirin ill into the 5% NaHC43}

Step 4} E a HCl3 from step (3) with 213 N H2SOa to recover ~i as drug

Remova! of ~e}~&orr~mon Substances:

3.

Ac~in~hen - Separation from basic drug:

Acetaminophen wifl often have #o been separated from basic drugs such as codeine and ~yd~ocodone. It can be extracted using the same procedure as tha# for mixtures of aspirin with basic drugs (see 2 abo~e).

4.

Caffeine — Separation from acidic or basic drug:

Caffeine is very soluble in chlorofarm and can be extracted from acidic or basic aqueous salutipns by washing 2-3 times with chioroform.

5.

Acid/Base Exfracion — General Cleanup:

Mar~y mixtures containing a basic drug car~ benefit from a generaf clear~up extraction to remove unwanted in#erferences. This is fundamentally the

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same procedure as 2 above without the bicarbonate wash.  
Step 1 } Dissolve sample in 213 N H<sub>2</sub>SO<sub>4</sub> and make the solution basic

with sodium Na<sub>2</sub>CO<sub>3</sub>

Step 2) Extract the sodium Na<sub>2</sub>CO<sub>3</sub> solution with CHCl<sub>3</sub>

Step 3) Extract the CHCl<sub>3</sub> from step (2) with 213 N H<sub>2</sub>SO<sub>4</sub> to recover  
the basic drug

- Oxidation of Cocaine Contaminants:

There are substances commonly encountered in cocaine samples that absorb in the UV range that may need to be removed or changed in order to obtain an accurate quantitation of cocaine purity. One common substance that is easily removed is caffeine which can be separated by the procedure outlined above.

The most common substances that are changed are benzoic acid and procaine (both indicated by yellow in the Van Urff's spot test), and benzoic acid. They are each chemically oxidized with a dilute potassium permanganate solution to produce nonabsorbing or weakly absorbing substances. Benzoic acid and procaine are primary aromatic amines and are oxidized to nitrates. Benzoic acid, an alkaloid present in the coca plant, has a double bond that is adjacent to an aromatic ring causing a strong absorption close to 275nm. This bond is oxidized by adding two -OH groups across the double bond to create a structure that has a weak absorption at 252nm.

Extraction for cocaine samples containing benzocaine, procaine, or cinchonine.

This is an Acid-Base extraction with addition of permanganate —

Step 1) Dissolve sample in 213 N H<sub>2</sub>SO<sub>4</sub>

Step 2) Add dropwise MnO<sub>4</sub> until the solution remains pink

Step 3) Add sodium Na<sub>2</sub>CO<sub>3</sub> to make solution basic

Step 4) Extract with petroleum ether (top layer) or CHCl<sub>3</sub> (bottom layer)

Step 5) Extract organic layer with 213 N H<sub>2</sub>SO<sub>4</sub>

Note: for quantitation accurately weigh sample in step 1 and accurately measure volume of 213 N H<sub>2</sub>SO<sub>4</sub> in step 5.

- Alkaline Diffusion or Conway Extraction

An effective means of separating amphetamine and methamphetamine from other substances is called alkaline diffusion and uses a Conway dish (hence the alternative name Conway extraction). This technique takes advantage of the volatile nature of the free base forms of amphetamine and methamphetamine in much the same way as the hanging drop microcrystalline technique. A strong base is used to neutralize the salt form of the amphetamine or methamphetamine and liberate the volatile free base form.

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S~b ect: 5e arations and Extractions Pa e 8 of 9

A strong acid\_..is used as \_a \_solvent to- collec: the ~rea ~asa am~ih~~ami~e or  
iiiet~iamphetamine which can then be quantitated.

Step 1) Place a weighed portion of the sampfe (20-25 mg} in the center well of a

Conway dish and add enough 10% NaOH to cover the sample.

Step 2) Pface approximately 3 ml of 213 N H<sub>2</sub>SO<sub>4</sub> in the midd~e ring.

Step 3) Add a few c}rops of dis#ilfed water to tf~e outer ring, place the lid o~er the

dis#~, and rotate the lid i~ the water to create a"seal".

Step 4) Alflow the dish to sit covered overnight.

Step 5) Carefully remove the lid and pipet out all of tF~e 213 N H<sub>2</sub>SO<sub>4</sub> pfacing it in a

5 ml graduated cylinder. Bring the voiume up to 5 ml with addit~onal 2/3 N  
HZSO~.

Step fi} Quantitate by UV as us~al.

~"~

~id

10°/a NaOH

Conway dish

#### PRACTICAL EXE

The trainee will~ i list of prac#ice ; performing various chemical  
ext~actions with the nce of the Trainer.

#### DOCUMENTATION

Th~ Trainer will review the proper Examination Sheet documerrtation for UVNIS results  
including quantitation (~urity) calculations (performed in Module 9). The Trainer will  
also review the proper case file documentation for extractian procedures.

#### STU DY GOALS

Understand pH measurement of acidic and basic solutions and how such  
solutions are ~repared. Understand the effect that a so4vent's pH can have on  
the solubility of solutes.

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Sub ect: Se arations and Extractions Ra e 9 of 9

~ Def~ne the terms "miscible" ar~d uimmiscibEe" and apply them to combinations of solvents.

- 

Know the general extraction scheme and apply it ta the separation of weak acidic, strong acidic, ~eutral, and hasic substances.

- Explain the principfe and use of a Conway extraction.
- Be able to perform variot~s separations and extractions for competency samples including CHCl3 washes, ~icarb washes, KMnOa extraction, and AcidIBase extractions.

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FTIR SPECTROPHOTQMETRY  
(to be initiaied when completed~

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A. Introduction  
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C. Baseline Correc '  
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Open OMNIC Icon  
Select HELP ~i GETTING STARTED -- BEGINNER'S GUIDE TO FTIR  
Select HE~P -- GETTING STARTED ~ SPECTROMETER TOUR

(the Spectrometer Tour module requires Disc 2: Spectrometer Tu#orials)

Select HELP ~ SAMPLING TECHNIQUES -- ATR SAMPLING TECHNIQUES  
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## HANBTON RAILCAMP PARTMENT CRIME LABORATORY MODULE 11.1

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Subject: IR / FTIR Spectroscopy Page 2 of 14

### OBJECTIVE

- To familiarize the trainee with the theory and application of infrared spectrophotometry in drug analysis.
- To familiarize the trainee with the FTIR spectrophotometry instrument and software used in the laboratory.
- To familiarize the trainee with the quality assurance procedures for the FTIR spectrophotometer.
- To make the trainee aware of the advantages, disadvantages, and limitations of FTIR spectrophotometry.

### DISCUSSION

- Introduction to Electromagnetic Radiation  
Gamma X-ray UV IR Microwave Radio  
High Energy Low Energy  
Transitions Nuclear Energy Vibrations Rotations  
Wavelength 10<sup>-2</sup> to 10<sup>3</sup> 10<sup>3</sup>  
(meters)  
Frequency 10<sup>24</sup> to 10<sup>14</sup> to 10<sup>1</sup> Hz  
(waves / sec)  
Wavenumber 10<sup>10</sup> to 10<sup>4</sup> to 10<sup>0</sup>  
(waves / cm)

The electromagnetic spectrum is a continuous range of radiation. The wavelength or frequency of the radiation defines the position of the electromagnetic spectrum. The shorter the wavelength, the higher the frequency, and the greater the energy of the radiation. The relationship between wavelength ( $\lambda$ ) and frequency ( $\nu$ ) is described by the equation

$C = \lambda \nu$ , where  $C$  is the velocity of radiation in a vacuum

This inverse relationship explains why shorter wavelength means greater frequency. To overcome this confusion the term wavenumber is sometimes used and is equal to  $1/\lambda$ , with units of cm<sup>-1</sup>.

Spectroscopy is the study of the interaction of electromagnetic radiation with matter.

This interaction of radiation with matter can cause redirection of the radiation and/or

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transitions between the energy levels of the atoms within molecules. A transition from a lower level to a higher level with transfer of energy from the radiation field to the atom or molecule is called absorption. A transition from a higher level to a lower level is called emission (fluorescence). Redirection of radiation is called scatter and may or may not occur with the transfer of energy.

When atoms or molecules absorb radiation, the incoming energy excites a quantized structure to a higher energy level. The type of excitation depends on the wavelength of the radiation. Electrons in their outer orbital (valence electrons) are promoted to higher orbitals by UV or Visible radiation. The absorption of infrared radiation causes vibrational excitation of molecules and low energy infrared or microwave radiation results in rotational excitation of molecules. It requires radiation of very short wavelengths in the X-ray region to excite inner orbital atomic electrons to a higher state. Radiation of even shorter wavelengths makes up the gamma ray region of the electromagnetic spectrum. It is transitions in atomic nuclei that result in the emission of this high-energy, penetrating radiation.

### Theory of IR Absorption

When a compound absorbs light in the visible and ultraviolet regions of the electromagnetic spectrum, electrons are promoted from lower-energy molecular orbitals to higher ones. Compounds may absorb electromagnetic energy in the infrared region of the spectrum. Infrared radiation does not have sufficient energy to cause the

excitation of electrons but it does cause bonds and groups of compounds to vibrate

about

the covalent bonds that connect them. The vibrations are quantized and as they occur, the compounds absorb infrared energy in particular regions of the spectrum. The infrared region of the electromagnetic spectrum encompasses radiation from 800 nm to 1,000,000 nm using units of nanometers, most modern infrared measurements are reported in terms of wavenumbers which was defined above as being equal to the inverse of cm<sup>-1</sup>. This is also convenient because wavenumbers are directly proportional to frequency and energy i.e. larger wavenumbers mean greater energy. Because the infrared region is so wide it is divided into three parts: the near IR from 12800 - 4000 cm<sup>-1</sup>, the mid IR from 4000 - 400 cm<sup>-1</sup>, and the far IR from 400 - 10 cm<sup>-1</sup>. It is the mid IR region which is most useful for analysis of drugs.

When a molecule is exposed to infrared radiation of a specific energy, the molecule can absorb the radiation by vibrating. At room temperature, the molecule sits in its ground electronic state and ground vibrational states. If the incoming infrared radiation has the appropriate energy, absorption occurs to excite the molecule to a particular higher vibrational state.

Molecules can undergo two types of vibrations, either stretching vibrations that involve changes in bond length or bending vibrations that involve changes in bond angles. To visualize these vibrations it is sometimes helpful to think of the covalent bonds between

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atoms as behaving like they were tiny springs connecting the atoms together. So

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Simple diatomic molecules like HCl, there is only one type of vibration possible and that is for the two atoms to move closer and further apart in a stretching motion. Three atom groups or more complex molecules can undergo a variety of stretching and bending vibrations which are summarized as follows:

Stretching Vibrations    Bending Vibrations  
(changes in bond length)    (changes in bond angles)  
Symmetric In-plane scissoring  
Asymmetric In-plane rocking

Out-of-plane wagging

Out-of-plane twisting

The infrared spectra of even relatively simple compounds have many absorption peaks. It can be shown that a nonlinear molecule has  $3N-6$  possible vibrational modes that can be responsible for the absorption of infrared radiation, while a linear molecule has  $3N-5$  possible vibrational modes. These are called fundamental modes and are the primary absorption bands which absorb infrared energy in the mid infrared range of  $4000 - 400 \text{ cm}^{-1}$ . To become excited, methane which has 5 atoms would have 9 possible fundamental absorption peaks and benzene would have

30.

Not all molecular vibrations result in the absorption of infrared energy, however. In order for a vibration to occur with the absorption of infrared energy, the dipole moment of the molecule must change as vibration occurs. Simple homonuclear diatomic molecules like nitrogen ( $\text{N}_2$ ), oxygen ( $\text{O}_2$ ), and hydrogen ( $\text{H}_2$ ) do not experience a change in dipole moment when they stretch so they do not exhibit infrared absorption. By contrast when heteronuclear diatomic molecules like HCl vibrate, the dipole moment changes and infrared absorption occurs. When the four hydrogens of methane vibrate symmetrically, there is no change in dipole moment so methane does not absorb infrared energy from symmetrical vibration. Symmetrical vibrations of the carbon-carbon single, double, and triple bonds of ethane, ethene, and ethyne do not result in the absorption of infrared radiation, either. Similar vibrational stretches in more complex molecules do absorb, but tend to be weak.

Vibrational absorption may occur outside the region measured by a particular infrared spectrophotometer and vibrational absorptions may occur so closely together that peaks fall on top of other peaks. These factors, together with the absence of absorptions because of vibrations that have no dipole moment change, cause infrared spectra to contain fewer peaks than the formulas  $3N - 6$  and  $3N - 5$  would predict.



However, other factors E~rsng about ever~ more absorption peaks. Overtone (harmonic) bands occur at integer multiples of fundamental absorption bands and result from the excitation of a ~ibration to a do~ble or higher frequency. Combination bands that are the sum or difference of two or more fundamental bands may also appear in infrared

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spectra. Both overtone and combination bands have weaker intensities than fundamental bands. It is also possible for absorptions to occur in the lower wavenumber (frequency) range due to molecular rotations.

The frequency (or wavenumber) of a given vibration and thus its location in an infrared spectrum can be related to two factors. These are the masses of the bonded atoms and the relative stiffness of the bond (bond strength). Again thinking of covalent bonds as springs connecting atoms together allows us to approximate the frequency of permitted vibrations by using Hooke's Law of Vibration which shows that the smaller the combined masses of the two atoms, the higher the frequency (light atoms vibrate at higher frequencies than heavier ones). The stronger the bond between two atoms, the more energy will be required to excite the stretching vibration. Triple bonds are stiffer (and vibrate at higher frequencies) than double bonds and double bonds are stiffer (and vibrate at higher frequencies) than single bonds. For example, the stretching frequencies of both a C-H bond and a C=O are higher than for a C-C bond.

An infrared spectrum is a plot of the vibrational and absorption bands present in a sample vs. the intensity of those bands over a range of radiation. Because infrared spectra contain so many peaks, the probability that two compounds will have the same infrared spectrum is exceedingly small. Optical isomers produce the same IR spectra.

- Instruments

Spectrophotometers are the devices that separate the numerous wavelengths within a range of radiation and allow for the absorption of the discrete wavelengths by components of interest to be studied independently. The graphical output from such an instrument is an absorption spectrum and it plots the absorption of radiation as a function of wavelength. As only certain transitions from a lower energy state to a higher energy level are allowed for atoms or molecules, an absorption spectrum can be a useful tool for identification of these substances.

#### Dispersive IR

Older IR spectrophotometers were very similar in design to UVVIS instruments. In fact they typically had the same five components including a radiation source, a sample compartment, a wavelength selector, a detector, and a readout device.

1. Source - creates radiant energy in the desired region. For infrared light a heat source (at high temperature) is typically used such as a Globar (piece of ceramic) or Nichrome wire

2. Sample Compartment - area where the sample is exposed to infrared light. There are various sampling devices and techniques available for gases, liquids, and solids each designed to maximize response while minimizing sample preparation. Examples include preparing KBr pellets from solids or using thin films of liquids between two salt crystal windows.

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The technique of ATR will be discussed in FTIR instruments.

3.

Monochromator — selects desired band of radiant energy via a diffraction grating. Many instruments place the monochromator after the sample compartment and immediately before the detector to minimize the effect of stray infrared light.

4.

Detector — device for measuring unabsorbed (transmitted) radiant energy having passed through the sample. Includes an amplifier to increase the signal from the detector. The preferred detector is composed of deuterated triglycine sulfate (DTGS) which is a pyroelectric detector that can convert infrared heat energy into electrical energy.

5.

Recorder — produces spectrum (graph of transmittance and absorbance vs. wavenumber).

As in VNIR spectroscopy there are both single-beam and double-beam dispersive IR instruments. The only important differences are that the design of the sample compartment in a double-beam instrument must accommodate a sample and reference area and that the double-beam system splits to alternately send the light from the sample beam and the reference to the monochromator. Whether the reference signal is collected separately in a single-beam or alternately in a double-beam instrument, it is tied with the sample signal to give transmittance or absorbance values at each wavelength.

FTIR Instruments

The primary difference between the IR instrument and a Fourier Transform IR (FTIR) instrument is that the monochromator is replaced with an interferometer. The interferometer is comprised of: a beamsplitter, a fixed mirror, and a moving mirror. Before discussion of the interferometer, it is helpful to define the terms constructive and destructive interference. Constructive interference occurs when two light beams are in phase so that their amplitudes add to give a light beam whose resultant is greater than the amplitude of either of the individual waves. When light beams are completely out of phase destructive interference occurs so that the combined amplitudes cancel each other out resulting in a light beam of zero amplitude.

To obtain an FTIR spectrum, the instrument's source generates light across the region of interest. Just as in the dispersive instrument. This light enters the interferometer and is split into two beams by the beamsplitter which directs the beams in two different directions at right angles. One beam travels to a fixed mirror and returns to the beamsplitter, while the other beam proceeds to a moving mirror (which varies the total pathlength of this beam) and returns to the beamsplitter. When the beams converge back at the beamsplitter, they recombine. If the two beams have traveled the same distance, then their pathlengths are the same (they are in-phase) and recombination is

fully constructive producing a single beam whose intensity is twice that of the source beam. If one of the beams travels  $\frac{1}{2}$  wavelength more or less than the other beam, they are completely out of phase and the amplitudes cancel each other out producing zero intensity.

zero intensity. Between these two extremes, a combination of constructive and

destructive interference takes place, and the resultant light beam intensity varies between being more or less than the two individual beams. The recombined beam passes through the sample which absorbs different wavelengths based on its chemical properties. The beam finally reaches the detector which measures the intensity of the recombined beam after exposure to the sample and sends the signal to a data processor.

In a dispersive IR instrument the intensity of light is measured as a function of wavelength. In an FTIR instrument the intensity of light is measured as a function of the path difference between the two light beams. This plot is called an interferogram. The mathematical process which converts an interferogram into a plot of intensity vs. wavenumber is called the Fourier Transform.

To put it simply, the interferometer is able to encode the entire coming from the

source without using a monochromator to separate it into wavelengths. This encoded beam is exposed to the sample and the Fourier used to decode the IR beam and produce the final absorption spectrum.

FTIR instruments include a He-Ne laser used as an internal calibration of the IR light. The second use for the laser position is the moving mirror so that the optical path difference can be accounted.

Figure 1---Laser Beam

IR beam •

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There are three primary advantages to the use of FTIR instruments. First, there is no need for a monochromator as all wavelengths of IR light are measured simultaneously. This is called the multiplex or Fellgett advantage. In practical terms it means that several scans can be taken in the same amount of time it would take to make one scan on a dispersive instrument. These multiple scans can be averaged together to improve the signal-to-noise ratio of the final spectrum. A second advantage is the throughput or Jacquinot advantage. Because the sample is exposed to all wavelengths at the same time, there is no need for slits to restrict the wavelength of light. The absence of slits means that all light energy or intensity reaches the sample and eventually the detector. This advantage also increases the signal-to-noise ratio over dispersive instruments. Finally, the use of a He-Ne laser for internal calibration leads to a high wavelength accuracy. This is known as the Connes' advantage. The disadvantages to FTIR are the same as those for dispersive IR. The sample

should be relatively pure as the resultant spectrum is absorption from all compounds present in the sample. For an accurate titration the sample and comparison should be in the same salt or base. This is also an advantage in determining salt and base forms of unknowns. While IR can distinguish stereoisomers like ephedrine and pseudoephedrine, it cannot distinguish optical diastereoisomers.

#### ~ ATR Sampling

An increasingly common sampling technique is known as attenuated total reflectance (ATR). It can be used to analyze solids or liquids with minimum if any sample preparation required. The ATR crystal is inserted into the path of the encoded IR beam after it exits the interferometer. Mirrors in the attachment direct the IR beam into a crystal which is in contact with the sample. A portion of the IR beam exits the crystal and enters the sample where it is absorbed (attenuated) at the appropriate

wavenumbers. The attenuated IR beam leaves the crystal and is directed out of the ATR apparatus to the detector and data processor for conversion into an absorption spectrum. See the diagram of a typical ATR apparatus below.

For detector parameter  
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To better understand the ATR technique it is useful to look more closely at the process involved. After the IR beam enters the ATR crystal, it is totally reflected off of the internal surface instead of passing directly through the crystal. This is because the crystal is cut at a 45 degree angle. The IR beam will continue to bounce off the top and bottom surfaces of the crystal until it hits the opposite side which is also cut at a 45 angle to allow the beam to finally exit the crystal. When the IR beam hits the top or bottom surface a portion of the beam actually leaves the crystal and is referred to as an evanescent wave. If sample is in contact with the crystal's top surface, the evanescent wave will interact with and be absorbed by the sample. If absorption occurs, the evanescent wave intensity will be decreased or attenuated.

How far the IR beam (more specifically, the evanescent wave) penetrates into the sample is called the depth of penetration or DP. One factor which affects the DP is

the refractive index (r.i.) of the crystal and of the sample. The phenomenon of light bending as it passes from a medium of one refractive index to a medium with another

refractive index is known as refraction. The refractive index of most organic substances is the same and can be taken as approximately 1.5. The refractive index of the crystal depends upon its composition. For example, crystals made of germanium

(Ge) have an r.i. of 4.0 while zinc selenide (ZnSe) has an r.i. of 2.5 and diamond crystals have an r.i. equal to 2.4. This is important because the DP goes down as the r.i. of the crystal goes up. This means that for a diamond crystal is larger than the DP for a Ge crystal. Of course, a diamond is more expensive, but it is also harder and more resistant to scratches than Ge or ZnSe.

Another factor affecting the depth of penetration is the wavenumber or energy of light. As the wavenumber or energy increases, the DP goes down. So 4000 cm<sup>-1</sup> light will penetrate further into the sample than 400 cm<sup>-1</sup> light. Stated another way, the absorption will be weaker at higher energy than at lower energy. This is a major difference between spectra with an ATR apparatus and transmission spectra which don't suffer from wavenumber dependent factors. Fortunately, ATR spectra can be corrected to look more like classical transmission spectra. This makes comparisons between spectra taken using these two techniques easier.

#### • Interpretation of Spectra

As already discussed, when a compound absorbs infrared radiation the chemical bonds in the compound will vibrate at characteristic frequencies. The functional groups present tend to absorb infrared radiation in the same frequency range regardless of the structure of the rest of the molecule. This means there is a correlation between the frequencies at which a molecule absorbs infrared radiation and its structure.

Tables of functional groups and their corresponding frequency ranges similar to the one below are widely available for use in determining which functional groups are present in molecular structures. Depending upon the reference source there can be variation in the ranges listed for functional groups. It can be seen from examination of these tables that most absorptions above the C=O stretching range of 1650 — 1760 cm<sup>-1</sup> are due to



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various vibrational stretches. The bands below this range, from approximately 1400 -

400  $\text{cm}^{-1}$  tend to be much closer together and result from some stretching vibrations as well as rotational energy changes within the molecule as a whole, but are primarily due to complex bending vibrations. Due to this complexity this region is less useful for identifying specific functional groups. However, these absorption bands typically are quite specific for individual molecules and thus this region of the IR is commonly referred to as the "fingerprint region".

Frequency ( $\text{cm}^{-1}$ ) Vibrational Group

3600 - 3200  $\text{cm}^{-1}$  O-H stretch (broad) alcohol, organic acids,

and water

3600 - 3500  $\text{cm}^{-1}$  O-H stretch (sharp) phenol, alcohol

3500 - 3300  $\text{cm}^{-1}$  N-H stretch (sharp)

3300 - 2900  $\text{cm}^{-1}$  C-H stretch (sharp)

3300  $\text{cm}^{-1}$  C-H alkyne

3100 - 3000  $\text{cm}^{-1}$  C-H alkene (aromatic ring)

3000 - 2960  $\text{cm}^{-1}$  C-H alkane

2400 - 2300  $\text{cm}^{-1}$  Carbon dioxide

2260 - 2210  $\text{cm}^{-1}$  C=N stretch, nitrile

2260 - 2190  $\text{cm}^{-1}$  C=C stretch alkene

1700 - 1690  $\text{cm}^{-1}$  C=O stretch

1780 - 1710  $\text{cm}^{-1}$  stretch carboxylic acids

1750 - 1735  $\text{cm}^{-1}$  C stretch esters

1740 - 1710  $\text{cm}^{-1}$  C=O stretch aldehydes

1730 - 1710  $\text{cm}^{-1}$  C=O stretch ketones

1690 - 1650  $\text{cm}^{-1}$  C=O stretch amides

1680 - 1640  $\text{cm}^{-1}$  C=C alkene stretch

1300 - 1000  $\text{cm}^{-1}$  C-O stretch

1400 - 400  $\text{cm}^{-1}$  Fingerprint region

The region of 3600 - 3200  $\text{cm}^{-1}$  is usually associated with NH and OH stretching vibrations. These bands also give direct evidence for hydrogen bonding. If an alcohol or phenol is present without hydrogen bonding, a sharp peak from the O-H absorption

occurs from 3650 ~ 3500  $\text{cm}^{-1}$ . Codeine base displays a sharp singlet near 3515  $\text{cm}^{-1}$  which is indicative of the OH group at position six in the codeine molecule. Increasing the presence of hydrogen bonding causes this sharp peak to be replaced by a broad band. Good examples of broad OH groups include the common sugars lactose and sucrose. This is also the region where the water molecule has strong broad absorption bands. Amines also give sharp peaks in this region arising from free N-H stretching vibrations. Primary amines give two sharp peaks and secondary amines give only one.

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The 3000 - 2700  $\text{cm}^{-1}$  region is associated with aliphatic C-H stretching; however, many

compounds common to drug cases are amine salts, which tend to obscure the information in this region. This brings up an important point to remember about IR spectrophotometry and that is its usefulness in determining salt forms. In fact it is one of the few analytical techniques which can identify the salt form of a substance. Primary amine salts (e.g. amphetamine HCl) show strong absorption between 3200 and 2800  $\text{cm}^{-1}$ . Secondary amine salts (e.g. methamphetamine HCl) exhibit strong multiple absorption bands between 3000 and 2700  $\text{cm}^{-1}$ . At still smaller wavenumbers, tertiary amine salts (e.g. cocaine HCl) absorb between 2700 and 2330  $\text{cm}^{-1}$ .

Carbonyl compounds absorb strongly within the 1760 — 1690  $\text{cm}^{-1}$  region. Ketones such as methadone show the carbonyl stretch near 1715  $\text{cm}^{-1}$  while aspirin's acid carbonyl stretch occurs near 1680  $\text{cm}^{-1}$ .

Esters have both a C=O carbonyl stretch and a C-O stretch around 1200 and 1240  $\text{cm}^{-1}$  respectively. Acetylcodeine, having a singlet ester group, shows 1735 and 1240

$\text{cm}^{-1}$  bands. On the other hand, heroin and cocaine show strong bands in the 1200-1300  $\text{cm}^{-1}$  region, indicative of two carbonyl absorptions, so exhibit the 1200  $\text{cm}^{-1}$  region band.

Although the 1200  $\text{cm}^{-1}$  region has been associated with esters, this region is also common to other C-O stretching band in the 1200  $\text{cm}^{-1}$  region usually indicates a C-O bond of some kind in the molecule. Methamphetamine shows an absence of bands in this region would be expected, while MDMA with a methylenedioxy group does have this region.

The 900 — 700  $\text{cm}^{-1}$  region is useful in determining the substitutions on aromatic benzene rings. Monosubstituted benzenes give two very strong peaks, one near 750  $\text{cm}^{-1}$  and the other near 700  $\text{cm}^{-1}$ . Toluene and the phenethylamines, amphetamine and methamphetamine, demonstrate these peaks. This region is also useful in distinguishing ortho-, meta-, and para-disubstitution on benzene. Ortho-substituted benzenes show an absorption peak arising from bending motions of the aromatic hydrogens between 700 and 770  $\text{cm}^{-1}$ . Meta-substituted benzenes show two peaks: one strong peak between 780 and 725  $\text{cm}^{-1}$  and one very strong peak between 750 and 810  $\text{cm}^{-1}$ . Para-substituted benzenes give a single very strong absorption between 790 and 840  $\text{cm}^{-1}$ .

Examination of an infrared spectrum can give very useful information into the structure of a compound by identifying functional groups present, but the identification of an unknown substance is best performed by comparing the absorption bands and their intensities over a full spectral range (from 4000 — 400  $\text{cm}^{-1}$ ) with that of known substances analyzed under the same conditions.

After a little experience the most commonly encountered drug samples can be recognized from their spectra by the analyst. Computer software programs exist which can assist the analyst in searching library collections of reference spectra for possible Effective date 05-04-09

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Subject: IR / FTIR Spectroscopy Page 42 of 14

matches. It is important that the analyst use automated library matching programs as an aid in making identifications and not rely on their results exclusively. Remember that the computer will always suggest a match. It is up to the analyst to determine whether the computer match is acceptable as the identification of an unknown substance.

When examining spectra for identification, contributions from interfering substances must also be taken into account. Atmospheric gases such as water vapor and carbon dioxide readily absorb in the IR region and steps are usually taken to minimize their contribution to sample spectra. One method is to collect a background spectrum under the same conditions as the sample spectrum. This not only removes the instrumental effects, but can remove water and carbon dioxide effects as long as the conditions for background and sample collection stay the same. If a sample contains liquid water it is best to try and dry it before collecting an IR spectrum as the broad absorption between 3700 — 3000 cm<sup>-1</sup> can mask or distort important absorptions in the region. If carbon dioxide is present in a sample scan, its asymmetric stretching around 2400 — 2300 cm<sup>-1</sup> does not usually interfere with other sample absorptions, it can however affect the quality of library searches, and it may "blow out" the carbon dioxide band before conducting such a search.

Because IR spectra from unknown sample will show absorption bands from all substances present, it may be necessary to sample before a satisfactory identification can be made. It is also possible to use library search routines to give an indication as to what other substances are present in a mixture other than those of interest. For example, lactose in a tablet, cocaine in a cocaine sample, or dimethyl sulfone with methamphetamine. What other substances are present may allow the analyst to use "subtraction" techniques to remove the contribution to the spectrum from these substances.

## PRACTICAL EXERCISES

- 

The train will be a list of practice samples for performing FTIR analysis.

- 

The Trainer demonstrates the use of the FTIR spectrophotometer to collect spectra from solid and liquid samples.

The Trainer will demonstrate the use of instrumental software to perform spectral adjustments including ATR corrections, CO<sub>2</sub> corrections, and spectral subtractions.

- The Trainer will demonstrate how to conduct identifications using both instrumental library searches and manual reference searches. The Trainer will demonstrate how to print results for inclusion in case files.

Once the trainee has completed the practice worksheet it will be reviewed with the Trainer.

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## DOCUMENTATION

The Trainer will review the proper labeling of FTIR spectra and the proper Examination Sheet documentation for FTIR results including base or salt form determinations.

## STUDY GOALS

- Know the general scanning range for IR spectra and the units commonly used for measurement of energy.
- Understand the principles of IR energy absorption by molecules and the types of transitions that take place including vibrational stretching (symmetric and asymmetric), vibrational bending (scissoring, twisting, wagging, and rocking) and molecular rotations.
- Be able to name and describe the three types of bands including fundamental, overtone, and combination bands.
- Know the principle components of both direct and Fourier transform instruments.
- Be familiar with the two main advantages FTIR spectrometers over dispersive instruments {"throughput" and "linear" advantages}.
- Know the two uses for a He-Ne laser in an FTIR instrument.
- Know the meaning of the terms constructive and destructive interference, and refraction.
- Be familiar with the basic principles of attenuated total reflectance (ATR) spectroscopy including the types of crystals commonly used {ZnSe, Ge, Diamond} and be able to define the term evanescent wave.
- Know the significance of air and water in IR spectroscopy.
- Be able to identify the functional group responsible for various infrared absorption bands including the following:  
N-H stretch (3300 cm<sup>-1</sup>),  
CO<sub>2</sub> stretch (2300-2400 cm<sup>-1</sup>),  
C-H stretch (2960-3300 cm<sup>-1</sup>),  
O-H stretch (3200-3600 cm<sup>-1</sup>),  
C=O stretch (1690-1760 cm<sup>-1</sup>)
- Be able to discuss the significance of the "fingerprint" region in an IR spectrum.

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Be able to discuss the variation observed in the C=O stretching region of the FTIR spectra for cocaine base and cocaine HCl.

~

Be able to match provided FTIR spectra to a list of possible substances including

the following:

Amoxicillin

Ampicillin

Carisaprodo!

Cocaine base

Cocaine HCl

GBL

GHB

Heroin HCl

Methamphetamine HCl

Toluene

~

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 Controlled Substances: Traini~g Guide Verslon 20a9  
 Sub'ect: Fourfer Transform\_I\_nfrared SFectrometr~Thermo Page 1 of 6  
 THERMO  
 FQUR~ER TRANSFORM INFRARED (FTIR) SPECTR~METRY  
 Instrument  
 Nicolet 47Q0 Se~ies FTIR spectrometer  
 Smart MtRacle ~iamond ATR Accessory  
 Software  
 OMNIC w~th Val-Q version 7.1  
 Startup

1.  
Turn on th~e monitor, pr~nter, and computer if not alre
2.  
Ensure that the instrument is on by checking t t t , Laser, and Source  
in~icator lights are green. T~e Scan indfc ash with each scan of  
the interferometer. The instrument power sh ay on at ail times. If it i~as  
been tuned o~f, tum it vn by pressing th s'ttch on the external power  
supp~y. Let it stabilize for at ~east 15 ' es ne hour for best results) before  
collecting spectra.
3.  
4~ the comp~ter desktop, open the by cfo~ble-ciicking the OMNIC icon.
4.  
If the ATR accessory is instaEled o tine analysis}, a window will appear to  
confirm #he proper Experiment et d. Choose "Smart MIRacle Accessory"  
from the drop-down menu t 4K,
5.  
The instrumen# will perf ia ostic tests, the results of which are indicated as  
"Bench Status" in th -up ght corner o~ the window. If #he indicator is a  
green check mark rometer has passed all of its diagnostic tests and is  
now ready # co ct c ra. If the indicator is a red X, the spectrometer has  
failed a diagn st ar~d requires corrective action before use. A message  
appears plai g the prvblem and alfows access to information about  
correcting i., e rcf the results in the logbook.
6.  
Check the inte al cfesiccant status by selecting Collect and Experiment Setup  
from the men~ toolbar. Select the Diagnostic tab, then Check Desiccant artd  
OK wher~ ~nished, R~cord the results in the logbook.
7.  
Align the spectrometer as follows:
  - 1.) While in Experimnt Setup, Select the Bench tab and check that Gain = 1.
  - 2.) Be sUre that there is no sample in the beam path. Selec# the Diagnost~c tab,  
record the Max interferogram value ir~ the logbook and then select Align.
  - 3.) A window appears advising that Benc~ Alignment is in pragress ar~d should  
take 2-3 minutes to complete. Record the Max interferogram val~e after  
alignment in the logbook and select OK.



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Subject: Fourier Transform Infrared Spectrometry Page 2 of 6  
Shutdown

1. Exit OMNIC by selecting the red X in the upper right corner of the window.
2. Turn off the monitor.  
Collecting Background and Sample Spectra  
Before collecting a spectrum, ensure that the OMNIC window is active, the correct Experiment Method is selected (Smart MTRAC Accessory for ATR experiments) and that the Bench Status indicator is a green check mark. See Startup Section for reference.

1. If a previous scan is displayed in the OMNIC window, clear it before starting a new scan by clicking the small gray X just above the Bench Status indicator.
2. To collect sample scans, select the "Col Smp" icon. A background is required before every sample scan, a prompt to collect background will appear. Select OK when ready.
3. When the background is complete a prompt for sample spectrum will appear. Select OK when ready.
4. When the sample scan is completed the spectrum title will appear. Overwrite the date and time with site (laboratory and exhibit number for example). Select OK.
5. Select YES to add the spectrum to the window.
6. Select OK if prompted for a window.
7. To save a file, select File AS from the toolbar.
8. Overwrite the default file name with the next data file name from the logbook and select SAVE. Record appropriate information in the logbook.

Data Analysis ~  
To perform a library search ~

1. Select "Library" icon from the toolbar. The parameters should read as follows:  
Search Libraries: FBI HPD IN-HOUSE (or whichever libraries are desired)  
Search Results: Configure search results button selected

Search type: Correlation  
Library: compounds with match values above: 0 (default)

Maximum number of compounds in list: 10 (default)  
Number of library spectra to display: 1 (default)  
Show match values selected

Search Regions tab Use full spectra! range selected  
NTE: This is not necessary with every run, since the parameters will not change from run to run.  
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2. Se~ect "Search" t0 r3~'ffOf~71 th@ 5@a'ifCFI.

To perform a spectrum subtraction

As FTIR spectrometry produces a cambinad s~ectrum for all components in a sample, the analyst may need to remove the contribu#ion from a major component to identi~y another component of interest. This is usually indicated from the search results where the first match accounts for some but not all of the sample peaks (ex. benzocaine with cocaine or suc~ase with amoxicillin). The analyst can use the Subtract function to remove these indicated substances and then perform a new search on the resultant spectrum as follows;

1.

After ~u~ning the sample, perForm a Search to det ine what should be subtracted. (Usually t~e first match will be a good pla start.) Close out of the search window and return ko the original sample sp ru

2.

Select "Lib Mgr" from the taolbar,

3.

Open "Search Libraries" and select the lib { -HOUSE, HR Georgia Drvg, etc.) which contains the spectrum to be .

4.

Click on the "Text Search" tab.

5.

Type in the name of the substance to be #ra .

6.

Double click an t~e substance to b s a

7.

Add to the same window as the ori l ple spectrum.

8.

C1ose ~ibrary Manager.

9. Select "Stack Spe" from the to ar eparate the two spectra.

'10. Click in the ~alf of the wind e sample spectrvn.

~ 1. Hold down the Control ke an c ick in the other hal~ of the window with the spectrum to i~e subtra re should be a message in the Information bar which says "Two sp se cted").

12. Selec# "Process" olbar and click on "Subtract~.

13.

Three spectr a ear: The original sample spectrum, the spectrum to be subtract and ult of the subtraction (this sF~ould be on the bottom).

'f4.

The subtr "or~ f or can be adjusted by scrolling up or down on the Factor button or~ the side of the screen. The Coarse and Finer buttons may be used to increase or decrease the fac#or.

15. Once satisfied with the results, click on "Add to new window" and perform a new Search.

To print a spectrum scan or library search

1.

Select Report and Template from the menu toolbar.

2.

The default should be "HPD TEMPLATE". If so, then select Close. If not, then highlight it and choose Select.

3.

Select the "Prev Rpt" icon and prompts will appear to enter the Lab #, then Analyst, and then a Description.

4.

The screen will display a preview of the report with the information entered. If the information is correct, select Print and Close. If the information is not correct, then select Close without printing and repeat steps 3 and 4.

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MODULE 1

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Subject: Fourier Transform Infrared Spectrometry - Thermo Page 4 of 6

Instrument Performance Check (performed quarterly or as needed) - - - - -

----- Follow

routine Startup procedures before performing an Instrument Performance Check.

1.

Remove the Smart MIRacle ATR accessory (refer to p. 8 of the ATR User's Guide). Install the standard sample holder and cover (refer to p. 28 of the FTIR User's Guide). A window will appear to confirm the accessory change and that the "Transmission E.S.P." Experimental Method has been loaded. Select OK. A Smart Accessory Test window will appear. A green check mark verifies the accessory change, select OK and continue.

2.

Allow the system to equilibrate for at least 15 minutes.

3.

Start Val-Q performance check by selecting Analyze a !-(- from the menu toolbar. The file path at the top of the screen should be: \documents and settings\omnicadmin\my documents\ftirvalidation.c . 's not, then select Open, locate and select this file.

4.

Select Options. The default parameters should be as follows:

Select "Show validation report at end of file" --

Select "Show test limits in validation report"

Select "Save spectra for validation"

Root filename should be "QAQC"

Select "Delay before collecting lysine spectrum: 9 minute"

Select OK.

5.

Select Validate and the prompt to clear the sample compartment select OK when ready.

6.

At the Peak sample prompt, open the cover to the sample area and insert the Serialized 1.0 mil polystyrene card into the sample holder, close and lock the cover, and select OK.

7.

At the Zeroing prompt, remove the 1.5 mil card and insert the Serialized 3.0 mil polystyrene card into the sample holder, close and lock the cover, and select OK.

8.

Upon completion, the "OMNIC Val-Q Report" screen will appear. If prompted, enter the serial number (AFZ0400253) and analyst initials. Select Print and then

Close.

9.

Print the Spectral window by clicking the right Display button and selecting Print.

10. Print the Results worksheet by clicking the left Display button and selecting Print.

11.

Select Save and check for the correct file name "ftirvalidation.csv". Select Save and Yes to replace and then Close.

12.

Record the instrument performance check results in the logbook and store the printouts in the appropriate location. The test results obtained by utilizing the Val-Q performance checks are compared to prior results to verify that the system is working consistently over time. If any problems occur or the report obtained indicates failure of one or more tests based on the given factory pass-fail range, effective date 04-09

consult the assigned analyst(s), the Varian User's Guide, or the FT-IR Operation Troubleshooting section of the FT-IR Spectrometer Validation handbook for potential causes and corrective recommendations. If these do not correct the problem, the instrument should be taken out of service until corrective action is taken.

13. When finished remove the 3.0 mil polystyrene card. Remove the standard sample holder and cover. Install the ATR accessory and select OK to confirm the "Smart MIRacle Accessory" Experimental Method. A Smart Accessory Test window will appear. A green check mark verifies the accessory change, select OK.

#### ATR Correction

In the ATR technique, the depth of penetration (that is, the effective pathlength) of the infrared beam varies as a function of the wavelength of light: longer wavelengths (lower frequencies) penetrate the sample more deeply than shorter wavelengths (higher frequencies). As a result, the bands at lower wavenumbers are much stronger than those at higher frequencies. This skewing of intensities causes problems when searching a sample spectrum against a library collected using standard transmission absorbance techniques, since they have different relative intensities and band positions.

The Experimental Method used by HP sampling includes a correction for this effect which multiplies the sample spectrum by a wavelength-dependent factor to adjust the relative band intensities. The spectrum has bands more like those in a typical absorbance spectrum and can visually be compared with absorbance spectra or searched against a library of reference spectra.

#### Carbon dioxide correction

In a typical FT experiment the sample spectrum is ratioed against a background spectrum that contains all of the spectral characteristics of the instrument. These characteristics include absorptions due to any atmospheric water vapor or carbon dioxide. Ratioing ensures that the sample spectrum contains information that is characteristic only of the sample.

Since sample and background spectra are collected separately, the water and carbon dioxide absorptions may not be exactly the same in both spectra. This can result in positive (or negative) peaks in the water (3,600 to 1,600  $\text{cm}^{-1}$ ) and carbon dioxide (2,350 and 668  $\text{cm}^{-1}$ ) regions of the ratioed sample spectrum. These residual peaks may cause problems when a spectrum is searched against a library.

To remove excess carbon dioxide contributions from a spectrum use the following steps  
For an already saved data file:

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Sub ect; ~ourier Transform Infrared S ectrdmet -Thermo Pa e 6 of B

1.

Select\_Region\_tool.{the.second\_icon in.the.lower\_left of screen). \_ \_ \_ \_ \_

2.

Using the cursor, point to where you want the region to start and press and hold down~ the mouse button.

3.

While F~olding down the mouse button, mo~e the pointer to where you want the region to end. Release mouse button.

4.

Select Process and Straight line to fi#l the highlighted region with a solid l~ne.

5.

Click Selsectlon too~ (the first icon in the lower fieft of scteen} to ttim off the Region taol.

Search Type Algorithms

Correiatlon

Normally gives the best results and is recammended for st applications. T~e algorithm removes any effect of offset in the u~k~own spect i~us eliminating the effects of baselin~ variation. This is the usual method ~sed ir~ H Lab.

~~

Absolute difference

Puts more weight on the small differences betw~ ~wn spectrum and iibrary spec#ra. This means that impuri#ies will ha~e a la n the s~arch results.

Squared difference

Emphasizes tl~e large peaks in the This algorithm may be used when iden#ifying a noisy spectrum.

Absolute derivative

Gi~es small peaks and pea i increased effect on the search results. The algorithm removes any ~ iff es etween the unknown and library spectra caused by an offset in t~e u knS m. This algorithm Es useful when you want to emphasize peak po ' ra er than peak intensities. This algorith#hm may be used when identifyinc~ spe ,r ith a tilted baseline.

Squared der~vative~°'

Emphasizes large peaks as well as peak shape. The algorithm removes any differences between the unknown and library spectra caused by an offset in the unkr~own spectrum. This algorithm works well with spectra of poor quality.

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Subject: Thin Layer Chromatography \_\_ Page 1 of 7

#### THIN-LAYER CHROMATOGRAPHY READING LIST

(to be initiated when completed)

- 1) A.C. Moffat editor, Clarke's Analysis of Drugs and Poisons, 3rd Edition, 2014.  
Ch. 27 — "Thin-Layer Chromatography"
- 2) S. Belt, Forensic Chemistry, 2006.

Ch. 4- "Thin-Layer Chromatography...", pp. 116-121

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## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 12

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Subject: Thin Layer Chromatography Page 2 of 7

### OBJECTIVE

~ To familiarize the trainee with the theory and application of thin layer

chromatography (TLC) in drug analysis.

- To familiarize the trainee with the preparative, quality control, storage, and proper handling procedures for TLC developing solvent systems and visualizing reagents.

- 

To make the trainee proficient in the use of TLC.

- 

To make the trainee aware of the advantages, disadvantages, and limitations of TLC.

### INTRODUCTION

Thin Layer Chromatography (TLC) is a quick and sensitive technique for the separation and identification of the components of a mixture or of individual substances. The technique is based on the affinity that a molecule has for a mobile liquid phase versus a solid stationary phase.

The solid stationary phase is typically made of glass, plastic, aluminum, or other media coated with a thin layer of a solvent. A small amount of the sample to be analyzed and a comparison standard are "spotted" on a line near the bottom of this plate. The TLC plate is then placed in a shallow pool of developing solvent within a closed chamber, ensuring that the solvent line is below the spot line. This solvent, or eluent, is the mobile phase and travels up the TLC plate by capillary action. As the eluent moves and the spots, equilibrium is established between the molecules in the mixture which are adsorbed on the solid, and the molecules which are in solution. Because the components will differ in solubility, strength of adsorption, and distance traveled up the plate. When the solvent has moved an appropriate distance (usually about 75-95%) up the plate, the plate is then removed from the chamber, the final solvent front location is recorded, the plate is dried, and the separated components of the mixture are visualized (detected).

The first two steps in performing thin layer chromatography are spotting and development, but results cannot be evaluated without visualization. Spraying the dried TLC plate after development with a chemical reagent such as iodoplatinate is a suitable way to visualize most organic drugs. In addition to this method, physical detection can also be used. Some pre-coated plates are available with fluorescent indicators which can be used to detect substances absorbing at a particular wavelength. Substances absorbing at this wavelength will contrast sharply by appearing dark while quenching the greenish-yellow fluorescing background.

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Under given conditions of temperature, solvent system, and type of adsorbent (solid phase), the chromatographic behavior of substances is described in terms of the R<sub>f</sub> value. This value is a characteristic of a particular substance and is described as the ratio of the distance traveled by the constituent to the distance traveled by the solvent. This can be expressed as follows:

$R_f = \frac{\text{distance traveled by substance from the origin}}{\text{distance traveled by the solvent front from the origin}}$

distance traveled by the solvent front from the origin line

TLC allows the presumptive identification of a substance when its R<sub>f</sub> is compared to and found to be the same as the R<sub>f</sub> of a standard. In practice the values do not have to be measured numerically but can be compared visually to have traveled the same distance from the origin line up the plate.

### PREPARING SAMPLES AND STANDARDS

Although there are numerous acceptable ways to run tests, a common example is described below. For other methodologies, refer to the literature for instructions.

#### 1. Prepare the TLC chamber

a. Obtain a beaker or other jar and a watch-glass or other covering for the chamber.

~  
~

b. Cover the floor of the chamber to about 1 cm from the bottom with the appropriate solvent.

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- c. Place a piece of filter paper into the chamber, cutting one side so that it stands level on the floor of the beaker and takes on the cylindrical shape of the chamber (it is recommended that the tank be lined on three sides with filter paper).
  - d. Place the cover over the chamber, to seal to prevent evaporation of the solvent and to allow the chamber to equilibrate.
2. Prepare the

- a. Obtain a suitable sized TLC plate to fit
  - b. Using a pencil and ruler if necessary, draw a line approximately 2 cm from the bottom of the plate.
3. Prepare and spot the standard and sample
- a. Obtain the appropriate standard(s) and sample(s) and dissolve a small amount of each substance in a small amount of solvent such as chloroform (usually 1 mg/ml)
  - b. Dip a clean capillary tube into the standard solution so that the liquid moves slightly upward.
  - c. Apply the standard directly on one side of the line previously drawn on the TLC plate. The spot should be no more than 4 mm in diameter or the resolution will be lost.
  - d. Repeat steps (b) and (c) for all sample(s) and any additional standard(s), leaving adequate spacing between spots.
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The original spot of sample was too concentrated.  
POSSIBLE SOLUTION  
Rerun the sample after diluting.  
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PROBLEM The spots appear smeared, resembling a convex object.

REASON(S) Compounds with acidic or basic groups may appear this way.

POSSIBLE SOLUTION Add several drops of ammonium hydroxide (amine) or acetic

acid (carboxylic acid) to the eluent for clearer results.

PROBLEM The spots appear smeared, resembling a concave object.

REASON(S) The system was likely disturbed during development.

POSSIBLE SOLUTION Rerun the sample, avoiding any disturbances of the system.

PROBLEM The solvent front does not run uniformly.

REASON(S) The silica gel has chips, dust on the plate or the edge of the plate is touching paper or sides of the chamber.

POSSIBLE SOLUTION Remove the silica gel and preventing interaction between the edges of the plate and any other object.

PROBLEM Many spots appear on the plate.

REASON(S) Contamination by other organic compounds such as dust, oils from hands, etc.

POSSIBLE SOLUTION Use gloves when performing TLC and prevent leaks or spills.

PROBLEM No spots appear on the plate.

REASON(S) Sample was too dilute, not enough sample to obtain a

positive result, no sample at all, or solvent line is at or above

spot line.

POSSIBLE SOLUTION Before attempting to rerun the sample, observe the TLC

plate under the UV lamp. If spots do not show up, attempt to rerun the TLC, ensuring that the sample is allowed to concentrate or, spot the sample several times in the same place, allowing any solvent to evaporate between spots (this will serve to concentrate your sample as well). If the solvent line was above the spot line, rerun the sample, making sure that the spot line is above the solvent front.

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Intentional

The Trainer will review appropriate sections of the CS-S-P (see Syllabus/Checklist) for performing TLC. In addition, the Trainer will review the reagent quality control procedures for TLC solvent systems and visualizing reagents including a review of the various recipes contained in the Reagent Logbook.

#### PRACTICAL EXERCISES

The trainee will receive a list of practice samples for performing TLC with the assistance of the Trainer.

#### DOCUMENTATION

The Trainer will  
TLC results.

#### STUDY GOALS

- Understand the Controlled Substances Act and the Controlled Substances Act.
- Understanding, developing, and using the effective date OS-18-Q9

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- --- GAS

## CHROMATOGRAPHY 1 MASS SPECTR~METRY READING LIST

~to be initialed when completed)

1) R. Saferstein editor, Forensic Science Handbook, Volumes 1, ~ 982.  
Ch. 3—"Forensic Appfications of Mass Spectrometry"

2) A.C. Moffat editor, CEar~ce's Analvsis of Drups and Poisons, 3`~ Editian, 2004.  
Ch. 28 — "Gas Chromatography"  
Ch. 2fi — "Mass Spectrometry"

3) J. Yinon, Forertsic Mass Spectrometrv. 1987.  
Ch. 1—"Mass Spectrometry of Cvmmonly Abuse~ugs"

4) CND Analytical: Series of Analy#icai Profiles ~~"  
Forensic and Analy#ica! CF~emis#ry of Cl d i henethylamir~~s  
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## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 13.1

Controlled Substances Training Guide Version 2009

Subject: Gas Chromatography / Mass Spectrometry Page 2 of 17

### OBJECTIVE

~ To familiarize the trainee with the theory and application of gas chromatography

{GC} in drug analysis.

- 

To familiarize the trainee with the GC instrumentation and software used in the laboratory.

- 

To familiarize the trainee with the theory and application of mass spectrometry (MS) in drug analysis.

- 

To familiarize the trainee with the MS instrumentation and software used in the laboratory. ~

### DISCUSSION

- Gas Chromatography

Gas chromatography (GC), like other forms of chromatography, is a method of separating mixtures. The separation is performed in a column containing the stationary phase, either solid or liquid, which is maintained at a defined temperature (isothermal), or temperature-programmed in an overall constant flow of carrier gas (mobile phase). When a mixture of substances is introduced at the inlet, each component is swept towards the detector and is partitioned between the stationary phase and the gas phase. Molecules with the greatest affinity for the stationary phase spend more time in that phase and consequently take longer to reach the detector. Depending on the type of detector, a signal is processed, and sent to an integrator or a computer. Each substance passing through the column will have a characteristic retention time which is defined as the time (typically in minutes) from injection to peak maximum at the detector. ~.

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Schematic of a gas chromatograph

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# HOUSTON POLEC~ DEPARTMENT CRIM~ LABORATORY M~DUL~ 13.1

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Subject: Gas Chromatography | Mass Spectrometry Page 3 of 17

A gas chromatograph consists of several basic components including the following:

1. Carrier gas -- acts as the mobile phase
2. Injection port — vaporizes the sample and introduces it into the instrument
3. Column — acts as the stationary phase
4. Oven — maintains the column at defined temperatures
5. Detector — detects the sample components as they elute from the column
6. Recorder — produces printout of data for interpretation

## Carrier Gas

The purpose of the carrier gas is to transport the sample through the column to the detector. Selecting the proper carrier gas is very important because it affects both column and detector performance. The velocity with which carrier gas is forced through the column will affect the separation efficiency and analysis time. If the velocity of the carrier gas is increased, then the analysis time will decrease, but the separation efficiency may also decrease. This effect can be seen in the Van Deemter plot below for the three most common carrier gases: nitrogen, helium, and hydrogen. In this plot, smaller plate height is a measure of better separation of components of a mixture. It can be seen that virtually the same minimum plate height is achieved with each gas. The difference arises in the optimum linear velocity. Although lower than for nitrogen, the curves for helium and hydrogen are flat enough that acceptable separation can occur over a wider range of linear velocity for helium and hydrogen than for nitrogen. Another factor that should be considered is safety. It is generally considered to be safer to use helium as the carrier gas than hydrogen. The purity of the carrier gas should be at least 99.995%.

Impurities such as oxygen or water can cause column and detector deterioration.

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Van Deemter plot for the three most common carrier gases H<sub>2</sub>, He, N<sub>2</sub>

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Subject: Gas Chromatography 1 Mass Spectrometry Page 4 of 17

Injection ~5µg

In most GC systems the sample is injected using a syringe through a septum-sealing silicone rubber septum into a glass liner within a metal block, where it is vaporized and swept onto the column by the carrier gas. The metal block is heated at a fixed temperature high enough to convert the liquid sample into a "plug" of vapor.

Most GC systems use capillary columns with internal diameters much less than one millimeter. To prevent overloading these columns, the sample volume produced in the injector port is reduced by using a split vent system. In such a technique, a 1-µl sample may be injected, but only 0.01 µl enters the capillary column with the remainder being vented out of the system. This split ratio is controlled by balancing the carrier gas flow rate to the vent flow rate. If the vent flow rate is adjusted to 25 ml/min with a corresponding column flow rate of 1 ml/min, the useful vent:column ratio will be

25:1. This means that 25 parts of the injection is vented and only 1 part is allowed to pass on to the column.

The split injection technique prevents column overload of a significant portion of the sample. For samples whose component concentration is in the parts-per-billion range (e.g. toxicology), splitless injection technique may be required. The splitless mode requires care in the selection of the medium and chromatographic parameters. It is also more difficult to set up and control than the split mode; however, it permits far greater sensitivity.

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Flow diagram af a sp~it injector Flow diagram of a splitfess injector  
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Sub ect: Gas Chromatouraphy / Mass Spectrarneiry\_ „\_, \_\_\_\_ \_” \_\_\_\_ ,\_ \_\_\_\_ \_ Pa~e 5 of 17

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Most columns in use today are capillary columns with an intemaf diameter ranging from

0.53 mm (wide bore) to 0.10 mm {Rarrow bore). They are usually cons#ructed of fused silica (a very high-puri~r) glass, which has a much h~gher degree of cross link~ng within the si~icon-oxygen mat~ix than does ordinary glass. Essentially fused silica columns are long, flexible, very thir~ glass tt~bes w~ose inner wall has been coated with a polymer liquid statior~ary pl~ase. A varie#y of func#iona~ groups can be blended i~to the polysiloxane polymer chain to pravide stationary phases of different polarity ar selectivity. (This corres~onds to partitioning gas-liquid chromatography as opposed to liquid-solid adsorption TLC.) As column intemal diameter decreases, column efficiency greatly increases. Unfortunately, it is easier to overload a narrow bore column which

reduces sample camponent separation negatir~g the benefit of smaller d~ameter. On the other hand, the wide bore columns tend to have long~r sis times. A good compromise is going with a 0.25 mm internal diameter column.

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The ~lm thickness of the liquid s#ationary phase ha e~ the retention time and resolution efficiency of the coumn. Al~ other thir~gs al, as the film thickness goes up, the retention time will increase as wel lecular weight compounds

should be analyzed on thick-film columns to in se e ime the compaunds spend in the stationary phase, allowing them to s p e. ' h molscular weight compouncls should bs analyzed or~ a thin-fifm co~umn. i duces the length of time the analytes

stay in the column. The typical range~l ~ckness is 0.1  $\mu\text{m}$  to 5.0  $\mu\text{m}$  with ~.25

$\mu\text{m}$  being a good compromise for mos g mples.

Capillary columns may range in nght from 15 meters to o~er 1 QO meters. Longer coumns provide more resoiv , b~t increase analysis time and column cost.

Doubling the columrt leng o i reases resolution by approximately 40%, but under isothermal condition analysis time. Using temperature programming is a more efficien~ way o c sin sample component resolution than increasing column length. Thirry m~,er co n are often a good choice for general drug analysis.

The final factor to~sider in selecting a calumn for gas chromatography is the

stationary liquid phase itself. The function of the stationary liquid phase is to separate the sample components into discrete peaks. Most drug samples are relatively non-polar so non-polar or slightly polar liquid phases are best. In addition, the liquid phase should have reasonable chemical and thermal stability. Over time the liquid phase will start to detach from the walls of the column. This is referred to as column bleed. As more and more of the liquid phase vaporizes and bleeds off of the column, separation efficiency decreases and retention time decreases. To minimize loss of the liquid phase, operational conditions should be maintained at 10 to 15 °C below the column's recommended upper temperature limit. High carrier gas pressures and solvent events will also degrade the column coatings over time.

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Chromatographic columns are coiled and held in a basket that is mounted inside of an oven. The column oven must be able to be rapidly heated and cooled. This requires a well-designed system to provide adequate air flow. In most designs, the air is blown past the heating coils, then through baffles that make up the inner wall of the oven, past the column, and back to the blower to be reheated and recirculated. Ovens are usually constructed of low-mass stainless steel. For temperature programming it is desirable to have a range of temperature program rates from 0.1 to 50 °C/min. Hold times anywhere within the program should be achievable.

As the temperature of the oven and hence of the column increases the retention times for sample components will decrease. This is a direct result of increased kinetic energy imparted to the sample molecules.

Detectors ~

A detector, located at the exit of the separation column, senses the presence of the individual components as they leave the column. The detector volume must be small to prevent the remixing of components separated in the column. The electrical analog output of the detector is amplified and converted to a digital signal and sent to a computer system. Some detectors are selective to the type of substances to which they respond and others are considered universal detectors because they respond to a wide range of substances. Examples include ~

1.

Flame Ionization Detector (FID): Responds to nearly all classes of compounds. Uses a hydrogen flame. As the effluent enters the flame, the total current that can be carried across it increases proportionally. The response is roughly proportional to the number of carbon atoms present. The response is lower if the compound contains a non-carbon atom.

2.

Electron Capture Detector (ECD): Highly sensitive to halogen, nitro groups, and carbonyl groups. Contains radioactive source.

3.

Nitrogen-Phosphorus Detector (NPD): Highly sensitive and specific to nitrogen and phosphorus. Useful in environmental work.

4.

Mass Spectrometer Detector (MSD): Considered a general detector because of its ability to detect and identify a wide range of compounds depending upon the type of analyzer present. This type of detector will be discussed in greater detail below.

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Recorder~

The electrical analog output of the detector is ampl~ed, converted to a digital sign~~ and ser~t to a computer. The computer can process, store and display the resul#ing chromatogram and analytical results. Such systems provide a continuous record of tt~e detector response versus time.

#### Method Development

The process of adjusting instrumentai conditions in a GC to optimize resolution {separation) of the components af a mixture is calfd method de~elopment. Of course selecting the rigF~t carrier gas and col~mn are the first steps in method developmen# and this process has already been discussed in detail.

Typically, once the carrier gas and column ~ave been sele d, e next step is to adjust the carrier gas pressure to optimize the linear ~elo ' rates. For he~ium carrier gas a lir~ear ~elocity around 3Q cmisec and 1.0 ml/min through the coiumn is a good starting point.

~  
Usually during a sample run, the method is d ne to keep all conditions the same with the exception oi tempera#ure. Th, ~ t p e may also be keep constant ~isothermal) during the sample run, but tt~ i vantage is that analysis time may be excessively iong ~f the sample contai nds with a wide range of molecular weights. Low moiecular weight com ur ~will efute fram the column quickly, while high molecular weight compounds~ ; several minutes to elute.

Temperature programming c n e best results of runs at clifferent temperat~res. The sample is injected int t G system with the cotumn temperature below that of the lowest-boiing co p e sample. The column temperature is ther~ raised at a preselected rate. ie peaks, representing low-boiling components, elu#e essentially as t wo 'ng an iso#thermal procedure. As the coiumn temperature increases, the hig oili components are forced thro~gh the coEumn faster than under fsothermaa condition .

One probEem with temperature programming is t~at the back-pressure increases with temperature and will reduce carrier fl~w if a#law controlfer is not used. Cofumn blesd at the higher #emperatures wilf also increase, resulting in an increasing baseline. For this reason t~e column should be well conditio~ed at the upper temperat~re lirn it before being used for sample analysis.

Drug ana#ysts need to have procedures available to process a wide range of substances. Ha~ing o~e GC methocl which car~ sepatate and identif~r all of the potential drugs iikely to be encountered is not usuafl~ {~ractical as such a method may need to be thirty minutes long per sample. Often the best solution is to have a number of inethods a~ailable which are a few minutes fong and are designed to separate arrd identify groups of compounds of similar molecular weight or which have similar GC response.

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For example, one method may be designed for the low molecular weight amphetamines, another method for mid-range compounds, and one for high molecular

weight steroids. The selection of the proper method would depend upon the results of prior testing.

Gas chromatographic techniques are sometimes sufficient for a chemist to presumptively identify the components in a mixture with a high degree of certainty. Such identifications are based on the fact that for a given column under fixed operating conditions a particular substance is eluted by a definite volume of carrier gas. When the carrier gas is flowing at a constant rate, the retention of the substance can be expressed in terms of the retention time. The retention time of the substance is then compared to the retention time, under the same chromatographic conditions, of a known substance. This is comparable to the comparison of  $R_f$  values in thin layer chromatography.

Methods such as mass spectrometry, infrared spectroscopy and nuclear magnetic resonance spectroscopy may be combined with GC to provide more detailed information about the identity of the substance detected by the GC.

### Mass Spectrometry

Mass spectrometry (MS) is an important analytical technique for the identification of chemical compounds. In spite of the fact that mass spectrometry cannot be regarded as a spectroscopic method there is no involvement of an electromagnetic spectrum i.e. IR or UV.

The mass spectrometer (MS) is only one kind of detector which can be coupled with separation techniques such as gas chromatography (GC/MS) or liquid chromatography (LC/MS). Immediately after the sample component molecules elute from the GC or LC column into the MS, they are bombarded with energy. This causes them to separate and form ions with a positive charge. Some of the bonds holding the molecule together are broken in the process, and the resulting

fragments may recombine or break up further to form more stable fragments. Because of natural laws governing the relative strengths of chemical bonds, a given compound will ionize, fragment, and rearrange reproducibly under a given set of conditions. The positively charged fragments or ions are separated, collected, and measured for mass and intensity.

The instrument produces a record known as a mass spectrum which is a representation of the intensity of the charged species and the mass to charge ratio ( $m/z$ ). The position and intensity of the  $m/z$  values is called a fragmentation pattern and provides qualitative information about the compound. Knowing the structure of the molecule, it is possible to predict the fragmentation pattern. Conversely, knowing the fragmentation pattern, a plausible structure of the original molecule can often be suggested. In addition, the technique can be used in determining molecular weights.

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Subject: Gas Chromatography 1 Mass Spectrometry Page 9 of 17

A mass spectrometer, regardless of the type or manufacturer, consists of several basic components including the following:

1. Sample inlet system — directs the sample into mass spectrometer
2. Ionizing source — receives the sample and produces ions
3. Mass analyzer — sorts the ions based upon their mass-to-charge ratio
4. Detector — produces a signal proportional to the number of ions striking it
- 5.

Recorder — produces printout of data for interpretation

EXHAUST ROUGH

PURGE

DIFFUSION

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ION DETECTOR

GC INLET SYSTEM SOURCE

MASS DATA

SPECTRUM ANALYSIS

Each of these processes use more than one method or procedure. What follows is a brief overview of the commonly used in conjunction with a GC system.

### Sample Inlet System

The major concern in interfacing a GC and an MS is the great pressure difference between the two systems. While GC systems are operated at atmospheric pressure or 760 Torr, MS systems are operated under a high vacuum at  $10^{-5}$  Torr. The reason for the vacuum in an MS system is to increase the mean free path of ions (i.e. the average distance an ion travels before it strikes something) between the source and the detector. Creating a vacuum in the MS removes almost all atmospheric molecules ( $O_2$ ,  $N_2$ ,  $CO_2$ , etc.) leaving predominantly sample ions and thereby decreasing the chance that an ion will collide with another molecule before hitting the detector. This increases the sensitivity by ensuring that more ions actually reach the detector. Capillary GC columns need only a rough mechanical pump to remove excess carrier gas combined with a diffusion pump to produce the high vacuum.

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The most widely used method of ionization is by electron impact (EI) in which the vaporized sample is bombarded with a stream of high-energy electrons. Due to electron-electron interactions, the molecules lose both the incoming electron and a bound electron. The resulting molecule is a positively charged cation (usually singly charged +1). The number of molecular ions initially formed depends on the energy of the incoming electrons, with higher energy electrons forming more positively charged molecular ions. If electrons of 70 eV are used, then the molecular ion will have an excess of energy which causes bonds to break up, or "fragment", into other ions, radicals, and neutral molecules. The masses of these fragments and the abundance of these fragments is dependant upon the starting molecules and is reproducible under the same operating conditions.

Ionization:  $ABC + e \rightarrow ABC^+ + 2e$

neutral 70 eV excited

molecule molecular ion

Fragmentation:  $ABC^+ \rightarrow AB^+$

$\rightarrow A^+ +$

$\rightarrow AB + \bullet$

$\rightarrow BC$  (loss of a neutral)

$\rightarrow + B$  (rearrangement)

$a^+$

Intensity

m/z ratio

In addition to electron impact ionization, chemical ionization (CI) may be used. In this technique a reagent gas (e.g. methane, ammonia) is introduced into the ionization chamber; therefore gas molecules are ionized by a high energy electron beam (100-150 eV) prior to the introduction of the sample. After the sample is introduced, it collides

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#### Subject: Gas Chromatography / Mass Spectrometry Page 11 of 17

with the ionized reagent gas. This process generally results in the production of fewer fragments than in EI. Quite often in CI, quasi-molecular ions ( $M+1$ ) are obtained, thereby making it easy for the determinations of the molecular weight of the compound. Having fewer fragments produced can also make the resultant spectra easier to interpret.

#### Mass Analyzers (Ion Separation)

A mass spectrometer is distinguished by the design of its mass analyzer. Two types include the quadrupole mass filter and the ion trap. The function of the mass analyzer is to sort and separate ions according to their mass to charge ratio. Quadrupole mass filters are composed of four symmetrically shaped rods. A field is set up in the space between the rods by the use of an RF field and a DC voltage which may be varied to allow ions of different  $m/z$  to reach the detector. As ions are created in the source they are directed into the space between the rods. The DC voltages of the rods and the RF field are set so that only ions of a certain mass will be able to reach the end of the rods and move on to the detector. The mass range is scanned stepwise so that the intensity of ions at each mass can be measured.

Ion traps consist of three electrodes: two endcap rods with a hyperbolic ring between them. In an ion trap all ions created over a given time period are trapped and then sequentially ejected from the ion trap to the detector. In this manner all ions are stored while mass analysis is performed, unlike the mass filter mode of operation where only one value of  $m/z$  is stored. The time during which ions are allowed into the trap, called the "ionization period", is set to maximize signal while minimizing collisions. The analyzer can collect ions over a period of time and then detect them all at once, which is a type of instrument with high sensitivity.

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Detectors

The beam of ions which have been separated by the mass analyzer is directed through a set of focusing lenses to a detector, Usually an electron multiplier. A traditional system

used an X-ray lens to detect the positively charged ions exiting the mass analyzer into the electron multiplier. Newer technology uses a high energy dynode which attracts the positive ions and emits electrons which then strike the electron multiplier. The electron multiplier generates a signal which is sent to the data processor.

#### Recorders

The electrical output of the detector is amplified, converted to a digital signal and sent to a computer. The computer can process, store, and display data generated as a mass spectral plot of the intensity of ions with a specific charge ratio over a selected range.

#### Tuning

Tuning of a mass spectrometer involves adjusting of instrumental parameters to maximize sensitivity and ensure that the assigned charge ratios are accurate. The most common substance using a mass spectrometer is perfluorotributylamine (PFTBA). This compound is a volatile liquid which is very stable and a necessary requirement for reproducibility, PFTBA has a molecular weight of 671 with a molecular formula of  $C_{12}F_{27}N$  fragments which are used to tune the MS are 69, 219, and 502. The MS set to maximize sensitivity across the mass range or more so at the high mass range to increase the likelihood of detected molecular ions. ~ \_

#### Interpretation

A GC/MS instrument provides an enormous amount of information, which must be presented in an easily interpreted form. Most instruments provide two types of printouts, the first is known as the total ion chromatogram (TIC) and the second consists of the mass spectra for each component separated in the sample mix.

After sample components have been separated by the GC column, they enter the MS and are ionized producing characteristic fragments which are detected by the electron multiplier. The TIC is a plot of the intensity of all ions detected at a certain time. When no sample components are eluting off the GC column, no ions are being produced and the TIC shows no response at that time. As sample components leave the column they are converted into ions which causes a peak to appear on the TIC corresponding to the amount of that component in the sample. The time at which sample peaks appear on the TIC is referred to as the retention time and is characteristic of that substance.

As mentioned before, the mass spectra are plots of intensity vs. a range of mass/charge ratio. In a typical GC/MS sample run a mass spectrum is produced for each retention

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analyst can look at the mass spectrum for that same retention time and see a breakdown of the intensities for the ions present. These individual mass spectra can be compared with known substances run under the same conditions or with reference or computer based libraries to make identifications. Just as with all computer generated comparisons, the analyst must remember that this is an aid to making identifications and not to be relied on exclusively. It is the analyst who must determine whether computer matches are acceptable as the identification of an unknown substance.

There are numerous references available which discuss the interpretation of mass spectra, and the trainee is advised to review these sources for an in-depth analysis of this topic. Examples of mass spectra produced by commonly encountered drugs are seen in the following table and can be used to illustrate various points.

The term base peak is defined as the most abundant ion in a spectrum. The term parent peak refers to the ion corresponding to the molecule. The parent peak may or may not be the ion with the highest mass as re, some fragments produced in the mass spectrometer may have a molecular weight less than the original molecule. It is also possible that the molecular ion may be so weak that none of it reaches the detector so that the true parent peak is not present (see the intermission below). For stable molecular ions the base peak and the parent peak can be the same ion (as in opiates like hydrocodone below).

In general aromatic compounds or functional groups tend to produce relatively stable fragments {see hydrocodone}. By contrast aliphatic compounds {like cocaine/isopropanol} fragment very easily and can produce mass spectra with more abundant low mass ions.

Some ions are frequent in various mass spectra and represent stable functional groups or cations. These include the 72 ion produced by promethazine and LSD.

Also, the 58 and 91 series are both seen in methamphetamine, phentermine, ephedrine, and pseudoephedrine.

The mass spectra for ephedrine and pseudoephedrine demonstrate that mass spectrometry is not capable of distinguishing stereoisomers. Some structural isomers like methamphetamine and phentermine may give mass spectra which require close inspection to distinguish them (note the ratios of the 115, 117, and 179 peaks in this case). Another pair of structural isomers includes LSD and LAMPA. Here the mass spectra are distinguished by the size of the 72 peak which is reproducibly more prominent in the spectrum of LSD than in that of LAMPA.

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MODULE 1~•1

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PRD-CT1CAL EXERCISE

- The trainee will receive a list of practice samples for performing GC/MS analysis.
  - The Trainer will discuss sample preparation techniques including choice of organic solvent, clean-up procedures, and approximate concentration of samples through the use of various standard mixes.
  - The Trainer will demonstrate the use of the available GC/MS systems to separate components of a mixture and identify those components by their individual mass spectra.
  - The Trainer will demonstrate how to conduct identifications using both instrumental library searches and manual reference sets. The Trainer will demonstrate how to print results for inclusion in case files.
  - The Trainer will demonstrate the use of instrument to perform spectral adjustments including background subtraction.
  - Once the trainee has completed the pretest sheet it will be reviewed with the Trainer.
  - The Trainer will review fragments commonly encountered molecules and the resulting mass spectra using files from the Training Guide Monographs.
- DOCUMENTATION
- The Trainer will review the labeling of GC/TIC printouts and MS spectra. The Trainer will also review the Examination Sheet documentation for GC/MS results.
- STUDY GOALS
- Know the principle components in a GC/MS system and the purpose of each including the following:
    - GC: carrier gas, injector, column in a temperature regulated oven
    - MS: ion source, mass separator (quadrupole or ion trap),  
  
electron multiplier detector, data analysis system
  - Know which factors can be adjusted to increase separation efficiency in a GC system.
  - Be able to define retention time.
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Know the effect that column degradation will have on separation efficiency and retention time.

- Know the effect that changing various GC parameters such as temperature, flow rate, column coating thickness, and column length will have on retention time.

- Be able to predict the most likely elution order of a mixture of substances using a non-polar GC column.

- Know the carrier gas used in the Controlled Substances Section MS systems and know the advantages of various gases based on efficiency and safety.

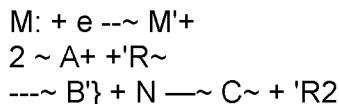
- Know the compound most commonly used to calibrate the mass axis of the mass spectrometer.

- Explain why an MSD system should be run under a .

- Be able to discuss electron impact ionization in system and how compound fragmentation occurs.

- Know the standard energy of electrons by the filament in an MSD source.

- Be able to identify the species which are detected in an electron impact mass spectrometer from the following equation:



- Know the types commonly used for Total Ion Chromatograms and Mass Spectra.

- Be able to identify variations in the fragmentation patterns of aliphatic vs. aromatic compounds.

- Define and identify the base peak and the parent peak from a provided mass spectrum.

- Be able to match provided GC/MS spectra to a list of possible substances including the following:

Acetaminophen  
Alprazolam  
Amphetamine

Carisoprodol  
Cinnamylcaine  
Cocaine  
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Cocaine  
Heroin  
Hydrocodone  
MDA  
MDMA  
Methamphetamine  
Monoacetylmorphine  
Phencyclidine  
Procaine  
Promethazine

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Subject: Gas Chromatography/Mass Spectrometry--Agilent, ^ Pa s of 4  
AGILENT

GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

Instrument

Agilent 6890 Network GC System

Agilent 5973 Network Mass Selective Detector

Software

Enhanced ChemStation G1701 DA version O.O. Q0.38 with upgrade D.p0.01

Startup

1.  
Turn on monitor.
2.  
Restart computer (recommended by manufacturer)
3.  
Open Instrument Control and Data Analysis e appropriate icons on the desktop.
4. After instrument initialization and loading, i or calibration.  
Calibration, Standards, and Tuning ( ly or as needed)
  1.  
Check the helium and make sure it is 300 psi. If it is, notify the assigned analyst(s).
  2.  
In the Instrument Control elect Instrument and highlight Perform MS Autotune.
  3.  
When the Select Tune up appears, check the appropriate tune and press OK. The first tune should be Standard Spectra Tune. Any additional tunes during the week should be Autotune.
  4.  
While the instrument is tuning, replace wash vials A and B with methanol and chloroform respectively, and replace the blank with chloroform.
  5.  
After the instrument performs the tune, examine the tune report using the following guidelines for acceptability:
    - The mass assignments shown in the upper "profile" part of the display should be within  $\pm 0.2$  amu of 69, 219, and 502.
    - The peak widths (PW) of these three peaks should be  $0.5 \pm 0.1$  amu.
    - The mass assignments shown in the lower "scan part" of the display should be within  $\pm 0.1$  amu of 69, 219, and 502.
    - The relative abundances should show that the peak at 69 amu is the largest. Relative to that peak, the one at 219 amu and the one at 502 amu should be in the range specified for the tune performed.

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Instrument methods are kept with the corresponding instrument As retention times  
Frequency change, a current copy will be  
kept with each instrument.

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Subject: Gas Chromatography Mass Spectrometry Appendix 2 of 4

- The isotope (Iso) mass assignments should each be 1 amu greater than the mass assignments of the parent peaks.
- The isotope (Iso) ratio figures (indicating the relative abundances of the naturally occurring isotopes) should be close to the theoretical values of 1.08 for m/z 69, 4.32 for m/z 219 and 10.09 for m/z 502.

If mass 28 is greater than mass 1\$, there may be an air leak somewhere in the system. Exceptions are when it is within 1 hour of venting, or during the first autotune after refilling the calibration vial.

If questions arise, simply refer to the instrument manual located on the desk next to the instrument or ask the appropriate analyst(s).

fi. To perform the daily blank and standard(s), select the Enhanced window.

7. Select Sequence from the menu and highlight it in the Log Table.
  8. When the Sample Log Table box opens, enter appropriate information and press OK.
  9. When the MSTopEnhanced window appears, in select Sequence and highlight Run.
  10. When the Start Sequence box appears, change the Data File Directory to reflect the appropriate date, ensure 1 Method is selected, and press Run Sequence.
  - ? 1. Record the appropriate information in the logbook after ensuring that the reports are acceptable.
- Shutdown

1. Turn off the machine.
  2. Never turn off the machine.
- Loading Blanks and samples when the instrument is not running
1. If the Defaults screen appears, press Cancel.
  - 2.

in the MStoPlEnhanced window, select Sequence and highlight Edit Sample Log.

3.

Ensure that all existing samples are finished running. If so, click Cut until all samples disappear.

4.

On the first line, begin entering the appropriate information and when finished press OK.

5.

When the MStoPlEnhanced window appears, select Sequence and highlight Run.

6.

When the Start Sequence box appears, ensure that the Data File Directory reflects the correct date, ensure that Full Method is selected, and press Run Sequence.

Effective date 05-18-08 .

Insument methods are kept with the corresponding Insument. As retention times frequently change, a current copy will be kept with each Insument.

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7. Make sure the information entered for all samples is also entered in the instrument logbook.

Loading Blanks and Samples when Instrument is Running

1.

Press Edit Sample Log Table in the MS Top Enhanced window to bring up the current sequence.

2.

When the Sample Log Table box appears, scroll to the first available line and enter information. When finished entering information press OK to allow the instrument to continue running.

3. Enter the appropriate information in the instrument logbook.

Data Analysts

7.

In Enhanced Data Analysis select the appropriate file to be analyzed.

2.

When the TIC window (window [2]) appears, right-click

the point in the

peak of interest where a mass spectrum is

TE: The peak can be

magnified by left-clicking and dragging around

to be magnified.

3.

Move the cursor to the Scan window (window

where the mass spectrum is

displayed). Right-click twice to search the library.

4.

When the next window appears (usually #24), compare the spectra.

5.

If the data is acceptable, label either the mass spectrum with the

star-darc retention time, if available

fi.

To do this, align the cursor with the notation R is to be made and press the left and right mouse buttons slowly,

7.

When the Annotate box appears, select a text position, enter the standard retention time for the sample and press Add.

8.

The identity of the peak is established by repeating steps five and 7.

9.

To print spectra/curves, select the Print icon from the menu.

10. When the Print appears, check Selected Window and press OK.

11. When the prompt appears, enter the appropriate window and press OK.

4ther

1.

Background subtraction

a.

With a TIC open in Data Analysis, find the area where a background subtraction is desired and right-click twice.

b.

Select File from the menu and highlight Subtract Background (BSB).

Multiple background subtractions can be performed by repeating the previous steps.

2.

Choosing the correct method

a. When entering samples in the Sample Log Table, to view a list of all Effective date 05-98-09

Instrument methods are kept with the corresponding instrument. As retention time and frequency change, a current copy will be kept with each instrument.

methods, press 5itirt +? simuitaneously whi~e the cursor is in the method box.

b.

When the Select Method box appears, highlight the appropriate methad and press OK. The selected method will now appear in the method box o~tf~e Sampte Log Table.

3. Changing search l~braries and searc~ parameters

a. In Data Analysis, select 5pectrum and highlight Select Library.

b.

When the Ltbrary Search Parameters ~ox appears, press Shift +? sim~~taneously while the cursor is in the appropriate box.

c.

When the Browse for Folder box appears, highi~light the appropriate search library (AAFS, HPD, a~d NIST98 are m frequently used} and press QK. The selected iibrary will now appear i appropriate Library Search Parameters box.

Effctive data 05-18-09Instrument

metriods are kept with the corresponding Instrumertt As retantfon times frequendy change, e airrent oopy wEll be keot with each InstrumeM.





When the tune is complete, compare the printout with previously accepted tunes and ensure that all values are within acceptable limits as given on the report. Store the printout in the appropriately marked drawer. If any problems occur, consult the assigned analyst(s) or the instrument manual stored next to the instrument.

10. To run the daily blank(s) and standard(s) check, select the Top icon, and then Batch Processing.

Effective date 05-18-09

Instrument methods are kept with the corresponding Instrument. As retention times frequently change, a current copy will be kept with each instrument.

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Subject: Gas Chromatography/MS Spectrometry Shimadzu Page 2 of 4

11. Go to the open File and open HPDBatchList4.

a.

In Row #1 under the Vial # column, type in or select "1" for the blank vial.

b.

Under Sample Name, type in Blank, Standard Name, and Initials (i.e. Blank COCSTD Lot# RR).

c.

Under Analysis Type, click on the right-hand side of the box and the Data Analysis Windows box will appear. Click 4FF Integration for Quantitative (IT), Quantitative Calculation (QT) and Click ON Integration for Quantitative TIC (1-T), Make Spectrum Process Table TIC (STT), and Similarity Search (LS).

d.

In Method File, click on the right-hand side of the box and choose the BLANK method.

e.

Under Data File, click on the right-hand side of the box and type in Blank 06/18/04 (appropriate date) and then OPEN or enter

f.

Under Report Output, check the print box ON.

g.

Under Report File, click on the right-hand side of the box and choose the Blank-C Report.

h.

In Row #2, repeat as Row #1, except for appropriate # for standard, Sample Name — i.e. COC R, Method File — DRUG method, Data File — i.e. COCSTD 0 18 appropriate date), and Report File — Report.

i.

In Row #3, repeat as Row #1 for Sample Name — Instrument Ready RR (appropriate initial, Method File — BLANK method, Data File — Blank2 06/18/04 (appropriate date), and Report File — Blank Report. .

12.

Delete the empty row at the end of the batch list. To do this, right click on that row, scroll down to delete Row, and select it.

13. Go to the Start icon and select it. This will start the scan.

14. After the scans are done and the results are acceptable, record the appropriate information in the logbook and store with the tune reports.

Shutdown

1.

If the HPDBatch~.ist is running, click on the batch list, go to Batch in the Toolbar, and scroll down to Daily shutdown after last batch. This will turn on the daily shutdown (to check if it is on, select Batch in the Toolbar again and observe a check mark on front of the Daily shutdown...). Turn off the monitor. Do not turn off or adjust the helium.

2.

If the instrument is not running, click on the batch list, go to Tools in the Toolbar, and scroll down to Daily Shutdown. The Daily Shutdown window will appear.

The settings in this box should read:

Under Line 1: SPL Temperature 250°C

Pressure 100 kPa

Total Flow 20.0 mL/min.

Column Temperature 150°C

MS Interface Temperature 250°C

Effective date 05-18-09

Instrument methods are kept with the corresponding Instrument. As retention times frequently change, a current copy will be kept with each Instrument.

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Under General: No boxes checked.  
Make any necessary changes then go to Shutdown and select it.

3. Turn off the monitor. Do not turn off or adjust the helium.

Loading Blanks and Samples when Instrument IS NOT Running

1.

In the HPDBatchList, delete the rows used previously to run the daily blank(s) and standard(s).

2.

Enter the appropriate information as follows:

a.

In Row # under the Vial # column, type in or select " " for the blank vial.

b.

In Sample Name, type in information to be printed on the report (for example, "Blank for lab # and initials")

c.

Under Analysis Type, click on the right-hand side box and the Data Analysis Windows box will appear. Click OFF the flag for Quantitative Calculation (keep ON Integration for Qualitative TIC (ILT), Make Spectra available TIC (STT), and Similarity Search (LS).

d.

In Method File, click on the right-hand side box and choose the BLANK method.

e.

Under Data File, type in the label file name (this is the sample data file # preceded by "8-")

f.

In Report Output, check the ON.

g.

Under Report File, click the right-hand side of the box and choose the Blank Report.

h.

In Row #2, repeat as row 1, except for Vial # "2" or appropriate # for sample, Sample enter your lab #, initials, and any other information to appear on the report, Method File — choose the appropriate, Data File — appropriate data file #, and Report file —

i.

Continue to alternate blank and sample rows as above until finished.

3.

Delete the row that appears at the end of the batch list. To do this, right click on that row, scroll down to Delete Row, and then select it.

4.

Go to the Start icon { ~ } in the Assistant Bar and select it. This will start the batch run.

#### Loading Blanks and Samples when Instrument IS Running

1.

Go to Assistant Bar and choose the Pause/Restart icon ( II ) and select it. This will pause the batch test and allow information to be entered into the list.

2.

Go to the last row # of the HPDBatchList and then right-click, scroll down to Add Row, and select it. For this added row, enter all information in the same manner as the above section (Loading Blanks and Samples when the Instrument IS NOT Running).

Effective date 05-15-09

Instrument methods are kept with the corresponding Instrument. As retention times frequently change, a current copy will be kept with each instrument.

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Subject: Gas Chromatograph/Mass Spectrometry—Shimadzu Page 4 of 4

3.

~~~~~ t~ empty row that d~,~,cars at the end of #~e ~atch !~s#. To ~o thi~, ~i^yy'~ click on that row, scroll down to Delete Row, and select it. .

4.

Go to the Pause/Restart icon (||) and click it OFF. This will prompt the batch list to continue the run.

5.

The instrument will not allow information to be entered when the last row is running. In this case, information will only be entered after the row (and batch) is complete. The information will then be entered as if the batch list is not running.

(above section — Loading Blanks and Samples when instrument FS NOT Running).

Inserting Blanks and Samples when the Instrument IS Running ~PRIORITIES~

1.

Go to the Pause/Restart icon (||) and select it. This will e the batch list and allow information to be entered into the list.

2.

Go to the row # you wish to insert a new row in row t click.

3.

Scroll down to insert Row, and select it. T~i row before the highlighted row.

4.

Input the information in the appropriate column of the section - Loading Blanks and Samples when the instrument IS NOT Running.

5.

Delete the empty row that appears in the batch list. To do this, right click on that row, scroll down to Del and select it.

fi. Go to the Pause/Restart icon (||) . it 4FF. This will prompt the batch list to continue the run.

Data Analysis

The instrument performs automatic data analysis and printing of major components from the Total Ion Chromatogram in the appropriate concentration range with reference spectra comparisons of the HPD library. The HPD library consists of spectra selected from the ISTD.

Effective Date 05-18-09

Instrument methods are kept with the corresponding Instrument. As retention times frequently change, a current copy will be kept with each Instrument.

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE ~3~

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Subject: Gas Chromatography/Mass Spectrometry —Varian page 1 of 4

iiARIAN

GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

Instrument#

Varian Chrompack CP-3800 Gas Chromatograph

Varian Saturn 2Q00 GC/MS

Software

Varian Saturn GC/MS Workstation System Control version 5.41

Startup

1.

Turn on monitor and restart computer (recommended procedure).

2.

Turn on monitor and computer (if computer is not running).

3.

Select Flow Pressure, press Up Cursor to verify.

4.

Set the Split Ratio to 20, and press Enter.

5.

On the desktop, select the System Control icon in the upper left corner to start the instrument software. Also, click the PN (PrintNow) icon for printing the tune results.

Calibration, Standards, and Tuning weekly or as needed)

1.

Check the helium pressure that it is ~300 psi. If not, notify the assigned analyst(s).

2.

Check the solvent was 0 ethanol) and fill it with methanol if it is less than half full.

3.

When System Control GC/MS #1 is loaded, select Windows and minimize 2000.

4.

Under Manual Control, it is a good idea to adjust the calibration gas. This is accomplished by clicking the Adjustments tab and clicking on Adjust Calibration Gas. After this, you will see blue bars adjusting in the display screen as the gas is equilibrating. Ideally, the bars should fill half of the screen, but this varies. If the gas needs calibrating, you must open the front compartment of the MS and adjust the CalGas knob in the appropriate direction until an acceptable level is attained. When finished, press Done and select the Auto Tune button.

5.

Check the Air/Water Check and FC43 Mass Calibration boxes, and the Start Auto Tune button. Once a week (or as needed) check the Electron Multiplier Tune box and proceed as usual with the tune.

6.

While these tunes are running, replace the blank with fresh solvent (chloroform).

When the tunes are complete, the message log at the bottom of the screen will read similar to the following example for an acceptable tune:

Effective data 05-18-09

Instrument methods are kept with the corresponding Instrument. As retention times frequently change, a current copy will be kept with each instrument.

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G6: 9 5: ~i5 H uio i une: ~iaRed

- 06: 95:05 Air/Water Check: Starfed
06:15:32 Air Check: Acceptable Level Found {2& Width: 1.4 m/z)
06:15:32 Water Check: Acceptable Level Found (19/18 Ratio: 9.3 %)
06:15:32 Air/Water Check: Completed - No Problems Found
06: 15:32 Auto Tune: Completed
06:95:32 Auto Tune: Completed
06:15:44 Auto Tune: Started
06:15:44 RF Full Scale Adj: Started
06: 95:54 RF Full Scale Adj: Centered on Mass 59 at 48.33 (Setting: 180)
06: 95: 59 RF Full Scale Adj: Centered on Mass 414 at 49.63 (Setting: 95 i~
06:16:04 RF Full Scale Adj: Centered on Mass 814 at 49.63 (Setting: 949)
06:16:44 RF Full Scale Adj: Setting is OK (Setting: ~49, ss: 614, Apex: 14.9)
06:96:04 RF Full Scale Adj: Completed
06: 96: 04 Multi-Point Mass Cal: Started
06: 96: 97 Multi-Point Mass Cal: Found Calibration 7.63
06:16:19 Multi-Point Mass Cal: Found Calibration at 68.10
06:16:24 Multi-Point Mass Cal: Found Calibration at 930.51
06:18:35 Multi-Point Mass Cal: Found Calibration at 213.55
06:18:54 Multi-Point Mass Cal: Found Calibration at 413.75
06:17:14 Multi-Point Mass Cal: Found Calibration Mass 464 at 463.76
06:17:37 Multi-Point Mass Cal: Found Calibration Mass 502 at 501.12
06: 98:04 Multi-Point Mass Cal: Found Calibration Mass 614 at 614.10
06: 18:04 Multi-Point Mass Cal: Calibration is OK (Slope: 6.28D, Std Dev: 0.052)
06:18:05 Multi-Point Mass Cal: Completed
06: 18:05 Auto Tune: Complete
The Multi-Point Mass Cal should be within +1- 0.5 of set mass. If not then retune.
7.
After the tune/ calibration is completed, select Hide Keypad on the left side of the menu bar and select Attributes from the drop-down menu in order to display the tune.
8.
Scroll down till tune results are displayed on the screen. Press the Alt and PrintScreen keys simultaneously and then Enter to print the results.
9.
Return the screen to its original state by selecting Show Keypad and Spectrum and Event Message Window from the drop-down menu and press the Acquisition button.
10.
In System Control, press the File Open icon in the menu and highlight Open SampleList.
11. When the new window appears, select the HPD.smp file and press Open.
12.

When the sample list appears, enter appropriate information for blanks and standards {if required}.

1-3.

Click the-Begin-button in the bottom left corner. and when the Begin Sample List box appears, select Browse, select HPD BLANK.mth and press Open and OK.

14. Enter the appropriate information in the instrument logbook after checking everything for acceptability.

Effective date 05-18-09

Instrument methods are kept with the corresponding instrument. As retention times frequently change, a current copy will be kept with each instrument.

MODULE 43.4

HOUSTON POLICE DEPARTMENT CREM~ LAB~RATORY

Version 2a09

Controlled Substances: Training Guide

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Subject: Gas Chromatography/MS Spectrometry—Varian
SFTutor

1.
Load the Shutdown method as you would any other sample. It is not necessary to actually activate the Shutdown method.
2. Turn off the monitor.
Loading Blanks and Samples when Instrument is not Running
1.
Select the Acquisition button in System Control.
2.
Select File from the menu and highlight Open SampleList.
3.
Select the HPD.smp file and press Open.
4.
If no samples are currently running, delete any and all entries.
5.
Double-click on the first box under Sample Name (this is the default parameters).
6.
Enter the next file number for the blank.
screen.
7.
Click on Sample Type and select Analysis from the
Click on Injection Notes and type in the appropriate notation.
- 8.
9.
Press Begin in the lower left corner.
Blank.mth method file,
10.
In the next window, press Browse, highlight
and press OK.
next available line, select
1.
For the next sample, under Sample Type
Activate Method from the dropdown menu and press OK.
12.
Click on Autolink, press Browse and enter the file number that corresponds to
p
3.

On the next available line enter

the sample.

Under Sample Type, select a sample from the pull down screen.

14.

as before in the Injection Notes box.

15. Enter the appropriate i

16. Enter the appropriate volume (e.g., 4 microliter) for a sample and at 1

17.

Leave the Inject

for blanks.

18. Enter the , proper formation in the instrument logbook.

Loading Blanks and samples when Instrument Is Running

Follow the instructions above, noting steps (11)–(18).

Data Analysis

1.

The instrument automatically generates a reference spectral library for each compound
the appropriate co

table.

2.

To perform a manual library search

Select the Chromatogram icon (4th from the left on the top toolbar).

a.

b. Click on the apex of the peak to be searched.

c. Click on the Library Searches icon on the 2nd toolbar near the top.

Effective date 05-18-09 Instrument

methods are kept with the corresponding instrument. As retention times frequently change,

a current library will be

kept with each instrument.

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- d. Click on the Peaks List box pull down window and select Best 3.
- e. Click on one of the search buttons (near the lower left corner).
- f. In the Target Spectrum window, click on Search.
- g. Label and print as always.
- h. Move the cursor to the next peak and search according to the above instructions.

Effective date 05-18-09

Instrument methods are kept with the corresponding Instrument. As retention times frequently change, a current copy will be kept with each instrument.

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Subject: Anabolic Steroids Page 10 of 15

ANABOLIC STEROID READING LIST
(to be initiated when completed)

1) D. M. Chiong, et. al. "The Analysis and Identification of Steroids", Journal of Forensic Sciences, 37(2), March 1992, pp 488-502.

2) K.K. Redda et. al., Cocaine, Marijuana: Designer Drugs: Chemistry, Pharmacology and Behavior, 1989.
Ch. 17 - "Uses and Abuses of Anabolic Steroids by Athletes"

3) CND Analytical • Series of Analytical Profiles
Analytical Profiles of the Anabolic Steroids Vol. 1

4) CND Analytical • Series of Analytical Profiles
Analytical Profiles of the Anabolic Steroid Substance Vol. 2

5) F. T. Noggle, Jr. "The Analysis of Anabolic presented at the

combined SAFLS-SVIAFLS-SAT Spring.
Effective date 05-18-09

MODULE 14

H~USTON POLICE DEPARTM~NT CRIME LA~ORATORY Version 2009

Contrvll~d Substa~ces Training Guide Pa e 2 of 5

Sub'ect: Anaboli Steroids

OBJ ECTIVE

To fami~arize the trainee with the class of drugs known as anabolic steroids and

the unique analytical challenges which they can present.

INTRODUCTION

Steroid hormones are lipids which play a major role in the physiology of mammalian systems. The steroid hormones include cholesterol, bile acids, Vitamin D, adrenocorticoids and the sex hormones. All the steroid structures contain the characteristic tetracyclic nucleus termed the cyclopentano-perhydrophenanthrene ring

system — a five-membered cyclopentane ring fused to a fully reduced phenanthrene ring

ings designated by

system. The steroid ring system is numbered by position and

letters as shown below:

H3 R

CH3a p 19 ~ ~ ~Z 1 17

Ha R CH3a~ R ~ y3 9 C D 1fi

H8 A 1p B 8 14 15

HB ,~

aa Haa 3 5

Haa Haa A~ 4 ~

Most rings are in the "chair" form and the methyl groups are assigned 18 and 19 (attached to 13 and 10 respectively). Functional groups at 3 and 17 differentiate the classification of steroids. Ring B can be cis or trans. Most cases involve a

eliminating the possibility of isomerism. Rings B-C

double bond at the 4 or 5

and C-D are always a 9 and 19 methyl groups are always above the plane, and hydrogen and substituent compared relative to the methyl groups (3 =

positions are always in the same position because of the trans

above). The 8, 14, 1

rings.

ANABOLIC ACTIVITY

Anabolic steroids are synthetic derivatives of testosterone, the male sex hormone secreted by the testes. The physiological actions of the anabolic steroids have been

classified as either androgenic or anabolic. Androgenic activity is primarily involved with the development and maintenance of masculine traits and the male reproductive system, whereas anabolic activity is thought to promote a more generalized growth of tissue by stimulating protein synthesis. Numerous compounds have been synthesized in an attempt to separate androgenic and anabolic properties. No such compound has yet been synthesized. Those compounds which demonstrate a high ratio of anabolic to androgenic activity are referred to as the anabolic steroids.

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Subject: Anabolic Steroids

Including veterinary drugs, there are over 100 anabolic steroids marketed worldwide. They can be subclassified as non-esters or 17-esters. The non-esters include testosterone, methyltestosterone, nortestosterone, boldenone, stanozolol, methandrostenolone, and others. The non-ester testosterone, nortestosterone, and boldenone are not effective unless injected, as they are rapidly metabolized by the liver (via oxidation of the C-17 hydroxyl group and reduction of the enone moiety in the A ring). Other non-ester steroids, including methyltestosterone, oxandrolone, and stanozolol are active upon oral administration due to the presence of an additional methyl or ethynyl substituent at the 17 position (not oxidized by the liver). The ester anabolic steroids include C-17 ester derivatives of testosterone, nortestosterone (nandrolone), boldenone and methandriol which are prepared as oil solutions for intramuscular injection.

17-OH ~

Testosterone Enanthate

Nandrolone Decanoate Oxymethalone

Effective date 05-18-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 14

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Subject: Anabolic Steroids Page 4 of 5

DISCUSSION

Control Status

The Anabolic Steroids Control Act was enacted by the Federal government in 1990 placing anabolic steroids in Schedule III of the Controlled Substances Act. In Texas the anabolic steroids are listed in Schedule III and Penalty Group 3 and are defined as

"any substance that is chemically or pharmacologically related to testosterone, other than an estrogen, progestin, or corticosteroid, and promotes muscle growth, including..." and in the Schedule III listing is added "(50) any salt, ester, or ether of a drug or substance described in this paragraph."

Analysis

form tablets, creams, Anabolic steroids are available in a variety of transdermal patches, water based suspensions, and injectables. Most packaging is

identification difficult at best. in a language other than English making pharmacist Even if packaging gives an indication as to the presence of an anabolic steroid, the widespread availability of counterfeit products makes this sort of information even more unreliable. ~~~

consulting information as to reagents to use Some literature sources provide chemical and expected results. Most chemical reagents use concentrated sulfuric acid

preparing or handling these reagents. Any as a solvent so care must be taken reagents other than those identified Frequently used would need to be quality checked with a standard prior to use. A steroid standard as long as the standard used gives a definite result e.g. Mandelin's Reagent gives a green color with codeine). Testosterone, methandrostenolone, commonly encountered in small round white tablets immediately turn red color with the addition of concentrated sulfuric acid (test in the Marquis test). UV spectroscopy is used as a presumptive test to indicate the possible presence of controlled substances. Unfortunately, most unknown samples are dissolved in 213 N H₂SO₄ which has a low solubility for anabolic steroids and may produce a bathochromic shift. The preferred solvent is methanol or ethanol. In these solvents a broad absorption from 240-245 nm can indicate an anabolic steroid such as testosterone which has a carbonyl at the 3 position and a double bond at the 4-5 position. Oxymethalone has a broad absorption in methanol at 282 nm. Another problem encountered in using UV as a test for anabolic steroids is that some common

preservatives absorb as well and can interfere with identification (e.g. benzyl alcohol gives a classic mono-substituted benzene absorption spectrum).

Thin layer chromatography (TLC) can be a useful secondary test for the presence of an anabolic steroid; however, the need to compare it with a known standard requires the analyst to already have an indication as to what the suspected anabolic steroid may be.

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Literature references __, __, suggest __, __, developing __, solvent __, systems __, and __ visualization

techniques. Methanol is again the preferred solvent for extracting the anabolic steroid from either tablet or oil.

GC/MS is the method of choice for analysis of anabolic steroids. In fact this is often the technique which gives the analyst the best indication of which anabolic steroid if any is present as it can identify ester forms such as testosterone enanthate. The samples may be extracted directly into methanol either from tablets or from an immiscible oil although the anabolic steroid stanozolol prefers ethanol (the analyst must make sure the ethanol is denatured 100% and not 95% as this would introduce water onto the GC column). If an oil preparation is miscible with methanol, then it must be diluted very heavily before running on the GC/MS. It is very easy to overload a GC column with anabolic steroids as they tend to be in high concentration preparations. It is better to get an indication of the presence of an anabolic steroid from a dilute preparation and have to re-run a more concentrated sample than it is to void a column and

spend several hours cleaning it out. The anabolic also require high temperature methods to elute from the GC column at greater than 400 amu as some of the esters have molecular weight over 400 (boldenone undecylenate MW = 452.7). Stanozolol usually better than other anabolic

steroids so if it is suspected to be in a sample, analyst may need to run more than one method to ensure its detection.

PRACTICAL EXERCISES

The trainee will receive a list of principles for the analysis of anabolic steroids to be completed with the assistance of the trainer.

DOCUMENTATION

The Trainer will review roster Examination Sheet and case file documentation for the analysis of anabolic steroids.

STUDY GOALS

- . Understand the difference between androgenic and anabolic activity in steroids.
- . Understand the control status of the anabolic steroids in Texas.
- . Understand the solubility properties of the common anabolic steroids and their

preference for alcohols over chloroform. Understand the unique analytical problems that can arise when identifying anabolic steroids.

- . Be able to match the chemical name for various anabolic steroids with the

provided structures.

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MARIHUANA READING LIST
(to be initialed when completed)

- 1) K.K. Redda et. al., Cocaine, Marijuana, Designer Drugs: Chemistry, Pharmacology, and Behavior, 1989.
Ch. 1—"Marijuana Pharmacokinetics and Pharmacodynamics"
- 2) C. G. Pitt, et. al. "The Specificity of the Duquenois Color Test for Marijuana and Hashish", Journal of Forensic Science, 17 (1972), pp. 693-700.
- 3) R. B. Hughes, et. al. "A Study of False Positives in the Chemical Identification of Marijuana", Journal of Forensic Science, 23(2), 1978, pp. 304-310.
- 4) K. Bailey, "The Value of the Duquenois Test is — A Survey", Journal of Forensic Science, 24 (1979), pp. 81
- 5) Various in-house articles discussing general marijuana topics including taxonomy, microscopic characteristics, spot testing, and testimony.

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Effective date 05-18-09

## HOUSTON POLICE DEPARTMENT CR1M~ LA80RATORY MODUL~ 15

Controlled Substances Training Guide Version 2009

Subject: Marijuana and THC Page 2 of 13

4BJECT~VE

To familiarize the trainee with the legal definitions, various forms, and analytical procedures for identifying marijuana, THC, and other cannabinoids.

To make the trainee proficient in the use of stereomicroscopes for identifying morphological characteristics of marijuana.

### INTRODUCTION AND CONTROL STATUS

Marijuana (spelled with an "h" in Texas instead of a "j") is considered to be the most commonly used illicit drug in the United States. It is a plant that grows wild throughout most of the tropical and temperate regions of the world and is a valuable, although illegal, cash crop in many regions of the U.S. and Mexico. In the Federal Controlled Substances Act marijuana is a Schedule I controlled drug but it is not recognized to have a legitimate medical use. As of 2009 states have passed medical marijuana laws which do recognize a medical marijuana as an appetite stimulant, pain reliever, nausea reducer, and triglyceride. While these states have passed legislation for the use of medical marijuana, these laws are in conflict with Federal laws. This means that individuals can still be prosecuted for possession and distribution of a Schedule I substance in Federal courts.

Marijuana contains cannabinoids that are unique to the plant, Cannabis sativa L. There are over 60 cannabinoids that have been identified and one of these, delta-9-tetrahydrocannabinol (THC), is believed to be responsible for most of the characteristic effects of marijuana. Research has resulted in the development and marketing of a synthetic THC called dronabinol. This product is approved by the FDA under the trade name Marinol, and is available by prescription for control of nausea and vomiting caused by chemotherapy and to stimulate appetite in patients. Federally, dronabinol has been placed in Schedule II recognizing its medical use and lower potential for abuse than marijuana. In the Texas Controlled Substances Act marijuana is defined as follows:

Marijuana means the plant Cannabis sativa L., whether growing or not, the seeds of that plant, and every compound, manufacture, salt, derivative, mixture, or preparation of that plant or its seeds. The term does not include:

{1} the resin extracted from a part of the plant or a compound, manufacture,

salt, derivative, mixture, or preparation of the resin;

{2} the mature stalks of the plant or fiber produced from the stalks;

{3} oil or cake made from the seeds of the plant;

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(4) a compound, manufacture, salt, derivative, mixture, preparation of the mature stalks, fiber, oil, or cake; or

(5) the sterilized seeds of the plant that are incapable of beginning germination. This definition applies strictly to the plant material and not to the active ingredient THC. It is also important to note that not all parts of the plant are controlled. Products such as clothing or rope made from hemp, the fibers of the mature stalks of the plant, are legal to possess or sell as hemp does not contain THC. The seeds of the marijuana plant are also free of THC (unless in contact with other plant material) and yield an oil which can be used as a substitute for linseed oil or in the making of soap. The waxy oil-cake from the inside of the seed is used in cattle feed or to make candles. In addition, the roots of the marijuana plant do not contain THC.

In Texas marijuana is listed as a Schedule I hallucinogenic substance but has its own penalty group for delivery or possession. This penalty group is unique in that the weight ranges are in English units of ounces and pounds instead of grams.

The dry leafy parts of marijuana are typically smoked in cigarettes, in cigars where the tobacco has been replaced with marijuana pipes. It can be cooked in food products and consumed orally although it takes longer to achieve the desired effects. Marijuana is generally considered a hallucinogen with the effects including a sense of euphoria, relaxation, changes of sensory perceptions including the slowing of time, and an increase in awareness.

The terms Bhang and Ganja refer to dried resinous flowering tops of marijuana which have been compressed. This is usually more potent than the dried leaves and stems because of the abundant THC-rich resin.

Thai sticks are prepared from marijuana leaves and/or flowering tops around a small wooden stick. The stick is smoked by breaking a piece off and transferring it to a pipe.

Sinsemilla is Spanish for seeds. This high potency marijuana is produced by thinning out or eliminating the male plants before they can pollinate the female plants. Such a step causes the female plant to produce more resin in its flowering buds.

The term hash or hashish refers to the THC-rich resinous material of the plant, which is collected, dried, and then compressed into a variety of forms, such as balls, cakes, or cookie-like sheets. Pieces are broken off, placed in pipes and smoked. Hash oil is produced by extracting the cannabinoids from plant material with a solvent such as alcohol. The color and odor of the resulting extract will vary, depending on the type of solvent used. The oil has a higher percentage of THC than marijuana or hash and is usually consumed by dipping a cigarette into the oil and smoking.

The terms hash, hashish, and hash oil are not defined in the Texas Controlled Substances Act, but are controlled as products which contain THC. The tetrahydro-  
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cannabinoids; including delta-9-THC, are listed in Texas as Schedule-I hallucinogenic substances and are placed in Penalty Group 2. This means that if a substance cannot be identified as marijuana but is found to contain THC, then it is controlled as a Penalty Group 2 substance and the weight would be reported in grams. Dronabinol, or synthetic THC, is listed separately as a Schedule III substance in Penalty Group 2 if it is "...in sesame oil and encapsulated in a soft gelatin capsule..." (see the Texas Controlled Substances Act for exact wording).

### BOTANICAL CHARACTERISTICS

It is now generally accepted that marijuana consists of one species with various varieties depending upon the environmental conditions under which it was grown. Such variables as origin of seed, local conditions of soil and climate, proximity of other plants during growth, selective breeding, and the length of the growing season all appear to

affect the final appearance of the mature plant.

The discipline concerned with the classification of living things is called taxonomy. It was Carl Linnaeus (sometimes called the father of botanical taxonomy) who in 1753 proposed the species name *Sativa*. The early trend in taxonomic works was to include Cannabis in the family (Urticaceae). In the late

1800's and early 1900's, most authorities placed it in the Moraceae family. The modern tendency places Cannabis with Humulus, the genus of the hop plant, in the family Cannabinaceae. Cannabis and Humulus are only two genera of the family Cannabinaceae. Marijuana is thus classified as follows:

Kingdom Plant  
Subkingdom phyta (seed bearing)  
Phylum Tracheophyta (vascular)  
Subphylum eudicotyledons (flower-like)  
Superclass (Dicotyledons)  
Class -Angiospermae (flowering plants)  
Subclass Dicotyledonae  
Order Urticales  
Family Cannabinaceae (Marijuana and Hops)  
Genus Cannabis  
Species *Sativa* L.

One of the points which has been debated within the scientific community and the court system is whether or not marijuana exists as other species, and if so, whether they are controlled. In 1785 La Marck proposed a separate species known as Cannabis indica which was smaller in stature and higher in THC content. In 1924, a botanist, Janischewsky, identified another species, Cannabis ruderalis which had adapted to northern climates. Most botanists now believe that Cannabis is monotypic, and that all others are merely varieties.

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Marijuana is a deciduous annual with a single growing season, where the leaves fall off at the end of the season. It is also dioecious i.e. the male and female flowers are borne on separate plants. Occasionally, however, male and female flowers occur on the same individual plant. This situation is described as monoecious. The ratio of male to female plants is reportedly influenced by exposure of seeds to ultraviolet light, air temperature, carbon monoxide concentration, the age of pollen and the stigma, and the nitrogen concentration in the soil. In general, the male plant is smaller and more sparse in foliage, while the female plant has a bushy appearance.

The stem of the plant is angular and sometimes hollow. It is covered with minute hairs curved upwards and appearing as if pressed against the stem. Each hair is formed from one cell and narrows rapidly from a stout base to an acute tip. These hairs are conical trichomes and are more commonly referred to as clothing hairs.

The plant sexes cannot be differentiated with certainty until they appear. The male (staminate) flower develops about three to four weeks after a female (pistillate) flower. The female flower consists of the bract, calyx and two slender pistils which are of an indifferent reddish-pink color. The flower has five sepals around the stamens. Once the pollen is disseminated to the female flower, the male flower soon begins to wither and die; meanwhile, the pollinated female continues to grow and produce seed. Usually, male plants are harvested earlier for fiber, or are eliminated as soon as they are recognized to permit unpollinated female plants to continue secreting resin to produce a greater yield.

The fruit technically contains a single seed with a hard shell tightly covered, the whole being regarded in practice as a "seed." The seed is round, smooth, about 2.5 to 5 mm long and 2 to 3.5 mm in diameter and mottled. It germinates within three to seven days, produces fifteen feet in height.

The leaves are composed of 5 to 13 separate leaflets with a palmate form. The thin and soft leaflets are lanceolate (i.e., approximately six times as long as broad and broad below the middle), with a narrow wedge-shaped base, a coarsely serrated edge and a long, drawn out pointed tip. The serrated edges are sharp and point toward the tip of the leaflet. The upper surfaces of the leaves bear unicellular, sharply pointed, curved, hairs with enlarged bases in which are located cystoliths of calcium carbonate. The shape of these cystolithic hairs (also conical trichomes) resemble the shape of a bear claw. This upper surface also carries the multicellular glandular hairs which appear when the plant is about to flower. The hairs have a shiny appearance and a sticky touch due to the exuding of resin. The lower epidermis bears clothing hairs which are longer and more slender than those of the upper surface and are without cystoliths. Cystolithic hairs are found on all parts of the plant including the roots, but are more abundant on the flowering plants and uppermost leaves. The mature seed is devoid of hairs.

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Cross-section of a Bract ~~~~~ Fr. ~~~~~ P! ~~~~~

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- (a) trichome hair
- (b) Large glandular hair with several cells in head and stalk
- (c) Head of one of the large glandular hairs
- (d) Small glandular hair with bicellular head and unicellular stalk
- (e) Thick walled conical trichomes (clothing hairs)
- (f) Large developing glandular hair
- (g) Stalk of a large glandular hair
- (h) Palisade cell
- (i) Cluster crystal
- (j) Parenchyma cell
- (Ec) Stomate

From The Botany and Chemistry of Cannabis, Joyce and Curry, 1970.  
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Characteristics of the Male Cannabis Plant  
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- A. mature male plant;
  - B. mature male flowering branch;
  - C. immature closed male flower;
  - D. mature open male flower with 5 sepals surrounding 5 anthers;
  - E. single anther and cross section of anther containing pollen
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Characteristics of the Female Cannabis Plant

- F. female flowering branch containing many flowers and bracts;
  - G. female flower with 2 projecting stigmas;
  - H. bractlet removed from around female flower,
  - I. female flower with bractlet removed showing ovary and 2 stigmas;
  - J. inside of bractlet;
  - K. outside of seed;
  - L. seed in longitudinal section showing embryo;
  - M. seed in cross section;
  - N. embryo removed from seed showing root and cotyledon
- Effective date 05-18-09

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CHEMICAL CHARACTERISTICS

There are at least 49 known chemicals that have been isolated from the plant *Cannabis sativa* L. Among the 419 chemicals are over 60 known cannabinoids. The male and female plants contain similar amounts of cannabinoids per fresh weight of plant material. At maturity (i.e., after flowering), male plants wither; meanwhile, female plants possess richer foliage and tops and consequently yield more cannabinoids per plant (weight) than the slender male plants.

The levo isomer of delta-9-tetrahydrocannabinol is the principal psychoactive ingredient of the marijuana plant. It has been shown that plants contain about the same amount of THC regardless of sex. Roots, large stems, and seeds contain little if any THC, while small stems and leaves are high in THC. Bracts (the upper leaves next to the flower), as well as the flowering tops (the buds), contain an abundance in secreting glands and, consequently, the highest amount of THC.

At

least four different numbering systems have been used to identify the various positions within the THC molecule. The "IUPAC" system and the "monoterpenoid" system are the most commonly used in present literature. The delta-9 designation is associated with the IUPAC system while the delta-9 designation comes from the monoterpenoid system. Note that delta refers to the position of the double bond.

Delta-9-THC is the most prominent psychoactive cannabinoid compound, and its concentration determines the potency of marijuana. In living plants, THC and most other cannabinoids are predominantly present in their "acidic" form (i.e., in the form of carboxylic acid derivatives) although to a lesser degree, in their "neutral" form.

Acidic and neutral cannabinoids have been extracted directly from marijuana preparations with or without heat. Acidic compounds, however, undergo decarboxylation quite readily. Decarboxylation is suspected to begin when marijuana is harvested and dried.

In addition to delta-9-THC, marijuana also contains delta-8-THC which is present in much lower concentration than delta-9-THC and is not as active. Other cannabinoids present include cannabidiol acid (CBDA), cannabinol (CBN), and cannabidiol (CBD) all of which are inactive.

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Section 11.1

Cannabidiol acid (CBDA) Cannabidiol (CBD)

Cannabinol (CBN) Tetrahydrocannabinol (THC)

### ANALYSIS

#### Botanical Examination

For the identification of marijuana, preservation of morphological characteristics is required. If a case consists of dried material, they are first dried to limit mold growth and remove excess moisture. An analysis of the plant should be removed prior to seed up the drying process. It will exhibit macroscopic characteristics which include the following:

#### Macroscopic Characteristics

1. Branches appear above each node with alternate pairs at 90° from each other.
  2. Leaf groups are compound, palmate (hand-like) in structure having an odd number of leaflets (5-11).
  3. Top side of leaf is darker than the bottom.
  4. Leaves are serrated, lance-shaped (lanceolate) and have pronounced veins running from a mid rib to tips of edges (pinnate venation) with branches at adjacent notches.
  5. Stalks are four-corner ridged, fluted (gives a square cross-section).
  6. Male flowering tops appear in sprays about 6" in length at the end of the stalk. They shed light green-yellow pollen profusely.
  7. Female flowering types — main twig arises immediately above the leaf; twigs arise alternately, branching further alternately, the latter bearing flowers and fruit; leaves differ in size from the rest of the plant.
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When marijuana is examined under a stereomicroscope, the analyst should observe more detailed characteristics including the following:

### Microscopic Characteristics

1.

Cystolithic hairs — "bear claw" shaped hairs; occur on upper, darker surface of the leaf, and have a calcium carbonate stone in the base which climbs into the interior of the hair; the larger the cystolith at the base of the hair, the shorter the hair.

2.

Cystolith — spheroidal calcium carbonate deposit at the base of the cystolithic hairs; can be seen without the claw as just a whitish mound.

3.

Clothing hairs (filamentous hairs, canonical trichomes) — long, thin hairs on the lower side of the leaf; also contain calcium carbonate, but not as much as the cystolithic hairs; more densely spaced than the cystoliths.

4.

Glandular hairs (resin glands) — multicellular hairs which secrete THC; shaped like clubs with flattened spherical heads of resin, unicellular or multicellular stalk; the stalk heads secrete resin. Usually abundant on the flowering tops of female plants; also found on seed bracts.

5.

Seeds — ovoid; green or brown mottled "turtle shell" appearance; approximate weight 10 mg each; from the hull, is ridged about the greatest circumference (dehiscence); inside is oily, coconut-like meat.

6.

Stems — "fluted" in appearance; covered with clotting hairs.

7.

Seed Bracts — leafy covering of seed; sticky; green, brown, or brown spotted. Often covered in glands when the plant is flowering.

8. Pistils — reddish brown fibers

### Chemical Screening

Chemical screening is used to test for the presence of marijuana, more specifically, for the cannabinoids produced by marijuana. Various literature articles have explored results of these tests and the possibility of obtaining false-positives. As a result of these studies, it appears that when used properly, the Duquenois-Levine test furnishes presumptive evidence for the presence of marijuana or a marijuana product. It can be performed directly on the plant substance or on an extract. The test will not only react with THC, but it will also react with CBD, and/or CBN to provide a blue-purple color via a phenol-aldehyde condensation reaction. Combining this chemical screening test with careful examination of the morphology of the sample plant material can serve as a reliable basis for the identification of

marihuana.

Thin Layer Chromatography

TLC may also be used to test a sample for the presence of THC or other cannabinoids for which comparison standards are available. One solvent system which can be used consists of the following chemicals in the noted proportions:

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Cyclohexane(50): Toluene(25): Acetone(95): Diethylamine(10)  
Once the TLC plate has been developed and dried it may be visualized with a solution of Fast Blue B salt. Positive results for the presence of cannabinoids from this test can be combined with a careful examination of the morphology of the sample plant material to serve as a reliable basis for the identification of marijuana.

#### Gas Chromatography / Mass Spectrometry

GC/MS may serve as a confirmatory test for the presence of THC or other cannabinoids. There may be case samples which indicate the presence of marijuana by morphological examination but do not yield positive results in chemical screening or TLC tests. In these cases organic extracts of plant material often be run on a GC/MS system to show the presence of cannabinoids like THC, or CBD. Other case samples such as hash, hash oil, or dronabinol gelcaps indicate marijuana plant material. In these situations GC/MS can be used in chemical screening or TLC to provide the basis for identification of THC.

DISCUSSION

The Trainer will review the appropriate sections of the Texas Controlled Substances Act regarding the status of marijuana, hash, hash oil, dronabinol, and the tetrahydrocannabinols.

The Trainer will review the appropriate sections of the CS-SOP (see

Syllabus/Checklist) for the analysis of marijuana and THC.

#### PRACTICAL EXERCISE 5

The trainee will receive practice samples to subject to microscopic examination

and chemical testing using the Duquenois-Levine test. The samples should

include the following (as available):  
casework marijuana comfrey leaves  
oregano nutmeg  
rosemary leaves "okonobong" leaves  
tobacco jimson weed  
coffee hogs  
passion flower yerbanis  
linden flower leaves patchouli oil  
damiana leaves sassafras\_oil

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- The Trainer will demonstrate the proper use of the stereomicroscopes available in the Controlled Substances Section.

- Once the trainee has completed the examination of the practice samples, the results will be reviewed with the Trainer.

DOCUMENTATION

The Trainer will review the proper Examination Sheet documentation for the analysis of marijuana. This review will include documentation of observed morphological characteristics on the Checklist for Marijuana Cases. The review will also cover the conversion of metric weights to English units.

STUDY GOALS

- Be able to define the term "Marijuana-based Texas Drug Laws."

Know which parts of the marijuana plant are used. Be able to explain what is meant by the terms "hemp" and "medical."

- Be able to explain the controlled status of "dronabinol", and "THC" based on the current Texas Drug Laws.

- Know the term used to describe substances unique to marijuana. Know the main psychoactive found in marijuana (the name not just the abbreviation).

Know that marijuana is considered to be an hallucinogen.

- Be able to identify name for various substances found in marijuana with the aid of a microscope.

- Be able to identify the various parts from the cross section of a bract from the fruiting cannabis plant.

- Be able to provide the complete botanical taxonomy of the marijuana plant.

- Be able to explain the proper way to conduct the chemical screening tests for the presence of chemicals unique to marijuana. Know the ingredients in the reagents used for these tests.

- Know which of the microscopic characteristics are required for the identification of marijuana.

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Subject: Evidence Handling

### OBJECTIVE

To familiarize the trainee with proper evidence handling procedures.

### DISCUSSION

The primary goal of the Controlled Substances Section is to provide quality analysis of evidence received for the presence of controlled substances, dangerous drugs, and other chemical substances as efficiently as possible utilizing available resources. This evidence is most commonly received from HPD officers or other law enforcement personnel. Once evidence has been received it must be handled in such a manner that . To accomplish this

no viable argument can be made as to its integrity and uniqueness and the Controlled

Substance Section procedures have been implemented by the Crime Laboratory and that

Controlled Substances Section to ensure that the integrity of the evidence is maintained and/or deteriorated

all evidence is protected from loss, cross-transfer, change.

"Chain of custody." This chain is

The total accounting of evidence items is known to the evidence since its acquisition by the evidence is made up of all individuals who have had custody of the evidence or "unaccountability" in the

law enforcement personnel. Because an  
chain of custody can seriously impair its admissibility in court, each individual  
assuming custody of the item must use the proper care, safekeeping, and  
preservation of the integrity of the evidence while it is under their control. The key to  
establishing this chain lies in the documentation of the transfer and possession  
of the evidence from one individual to the next in the normal course of business. This  
documentation consists of a variety of receipts or other forms, whereby a transfer of  
possession is recorded.

The general flow (documented through the chain of custody) of a piece of controlled  
substance evidence through the HPD Laboratory takes the following course, i.e. the  
"normal course of business". The evidence is received by Centralized Evidence  
Receiving (CER) personnel either directly from law enforcement personnel or indirectly  
through the offsite lock boxes. When CER personnel receive the evidence it is checked  
to ensure it is properly packaged, sealed, and documented. In order to ensure that the  
integrity of the evidence submitted is maintained, it is accepted only if the evidence  
container, generally a Houston Police Department Evidence Envelope, is properly  
sealed. It must be sealed in such a manner that there is no possibility that the package  
contents can be removed, altered or a substitution made without the seal being  
obviously disturbed. The actual seal itself must be initialed or otherwise marked to  
document the person sealing the evidence. In the event that evidence is received  
without a proper seal, it is subjected to remedial seal and inventory. If inventoried, a  
proper seal will be placed on the container. The container must also be marked with a  
unique identifier. This will ensure that all references are to this particular evidence and  
that no substitution could have occurred. After being received by CER personnel,

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from the Laboratory.

When evidence is assigned to an analyst, he/she will receive that evidence from CER and the transfer will be documented at that time as part of the chain of custody. The analyst will examine the evidence container(s) to ensure that proper seal(s) are in place and that unique identifiers are present on each container. The analyst will also mark each evidence container with his/her initials. The analyst should examine case associated documentation to see if latent print examinations are requested. If so, the analyst should take extra care when handling the evidence to maximize preservation of possible latent prints. After receipt of evidence the analyst should open any container(s) in a manner that preserves the existing seal whenever possible and immediately inventory the contents comparing them with the evidence description provided by the original submitting personnel. Any discrepancies should be noted and handled according to Section and Laboratory procedures. ~+

It is the analyst's responsibility to maintain the integrity of the evidence at all times while in his/her custody. Only one case is to be examined at a time to prevent cross-transfer. All cases within an analyst's care are secured in a limited access area when the analyst leaves the work area. ~~~

All exhibits contained within a case should be labeled with the analyst's initials and the unique case identifier. The analyst may see evidence such as loose tablets or

a broken glass pipe into another \_ in which is labeled with the unique case identifier and the analyst's initials and a note that it was placed in this container by the analyst. .

Each time evidence changes hands within the Laboratory, the transfer must be documented at that time in the chain of custody. The transfer of evidence to another division or a cut out of the Crime Laboratory (such as Latent Prints, the Property Room, etc.) is documented not only as part of the chain of custody, but also as a supplement to the case in OLO.

After the analysis has been completed, the analyst will seal all evidence containers marking the seal with his/her initials and the date. The evidence will then be returned to CER for long term storage.

If evidence is released for court, this will be done by CER personnel. It is recommended that the case analyst or other Controlled Substances Section personnel be present to view the release. This transfer is documented in the case file as part of the chain of custody, but does not need to be supplemented in OLO.

The Trainer will review appropriate sections of the CS-SOP and the Crime Laboratory Division Quality Assurance and Standard Operating Procedures Manual regarding evidence handling (see Syllabus/Checklist).

Effective date 06-08-09

HQUSTON POLICE QEPARTMENT CRIME LABORATORY MODULE 16  
Controlled Substances Training Guide Version 2009  
Sub'ect: E~vidence Handlin ~a e 3 of 3  
P~~T~~~L E~~RC~~~S

- 

The trainee wi~l have t~e opportu~ity to observe t~e receiving of evidence by the Centra~~zed Evidence Receiving Saction~ (CER} from various lock box~s.

- 

Th~ tratnee will observe the procedures for receiving and releasing control~ed substance evidence by the Trainer or designee.

- The trainee will ha~e the opportunity to demonstrate the proper procedures for recei~ing and releasing cor~#rolled subs#ance evidence d+~~ing analysis ~erformed by #he trainee and monitored by the Trainer or desigr~ee.

DOCUMENTA'

The Trainer wil documentation  
for receipt and ~isions such as  
Latent Prints ai  
STUDY G~AL~

- 

Be able s its impvrtance  
in casern

- Be able ~ce #o the HPD  
Cr9me L~  
. Be able

- Be able

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Be able ~f an anaEyst.

Be able to discuss ~ow to handle evidence which needs a fatent prin#  
examination.

Be able to discuss how to handle evidence which needs ko be transferred ta the  
HPD Property Room.

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HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 17  
Controlled Substances: Training Guide Version 2009  
Subject: Analysis Guidelines Page 1 of 4

#### ANALYSIS GUIDELINES READING LIST

(to be initialed when completed)

1) R. Saferstein editor, Forensic Science Handbook, Vol. 2, 2nd Ed., 2005.

Ch. 4—"Forensic Identification of Illicit Drugs"

2) SWGORD Recommendations, 2nd ed. "Part III B- Methods of Analysis/Drug

Identification", February, 2006,

effective as of 2/1/06

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 17  
Controlled Substances: Trafficking Guide Version 2009  
Subsection: Analysis Guidelines page 2 of 4  
OBJECTIVE

To familiarize the trainee with various analytical schemes for processing

controlled substance evidence.

DISCUSSION

Forensic science has been defined as the application of scientific principles to answer questions of interest to a legal system. The American Board of Criminalistics defines criminalistics as that profession and scientific discipline directed to the recognition, identification, individualization, and evaluation of physical evidence by application of the physical and natural sciences to law-science matters. Forensic chemistry can be thought of as a marriage between analytical chemistry and criminal justice where the needs of both parties must be met. In choosing the proper method of analysis for a particular case, a forensic analyst must consider factors: the needs of an investigator, what charges if any are being filed, sensitivity of tests in the scientific community, use of available resources, etc.

A controlled substance analyst is tasked with determining whether evidence submitted to the Laboratory is a controlled substance, a prescription drug, or other substance of interest. The analytical tests available to achieve this goal are generally considered to be presumptive or confirmatory in nature. Presumptive tests give an indication as to what substances may be present. Presumptive tests are simple and quick to perform and in spite of their lack of uniqueness can give very good indications as to whether substances of interest are present. Confirmatory test results are the most specific for a particular substance and are therefore considered the most discriminating tests available. In identifying unknown substances the analyst may need to determine the weight or purity of these substances. These tasks may or may not be included as part of the analytical scheme, but must be considered when choosing tests to perform. For example, one of the tests used for identification may also be able to determine purity requested.

The actual protocol for identification of an unknown substance is specified in the Controlled Substances Section SOP. It requires that a minimum of two different positive test results be obtained with at least one of the positive tests being a confirmatory GC/MS or FTIR. On the other hand knowing when to conclude that no substance of interest is present can be more difficult than knowing when sufficient work has been performed to make an identification. The best approach is to choose tests which will reasonably preclude the most common controlled substances or dangerous drugs from being present. Knowing the limitations of analytical tests is crucial. For example, negative chemical screening tests and UV spectrophotometry on a white tablet would not be sufficient to eliminate the possibility of the dangerous drug carisopropol from being present. When in doubt the opinion of a second qualified analyst can be very helpful.

Effective date 08-08-09

## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 17

Controlled Substances: Training Guide Version 2409

Subject: Analysis Guidelines Page 3 of 4

There are various analytical schemes or approaches used in determining whether a controlled substance, dangerous drug, or other substance of interest is present in a case submitted for analysis. As no analytical scheme can account for all scenarios, the analyst must use these schemes as a guideline and rely on experience, best judgement, and possible advice from more experienced analysts when forming conclusions in some cases.

Visual examination of an item of evidence is the first step towards making an identification. The analyst should determine if the item is a solid or liquid or plant material. What is the texture and consistency of the substance? Is it a powder or crumbly chunk substance? Does the material have a waxy consistency? Are there markings on the item which indicate legitimate manufacture or clandestine origin? What is the viscosity of the liquid? The color and odor of the material can be good indications as to the possible identity of the substance. For example, a brown powder with the odor of vinegar may contain heroin. A yellowish fluid with the odor of ether should be examined for the presence of phencyclidine. The analyst considers how much of the sample is available for testing. If a very small sample is present, the analyst will want to proceed with nondestructive and sensitive tests to minimize the amount of sample used.

Following visual examination there are likely scenarios. In the first scenario, the analyst has an idea of what substance is present and there is sufficient sample available for testing. The first the analyst performs should be one which would support this idea. If the results of this test are positive, then the analyst can select an appropriate confirmatory test. For example, if a white powder is suspected of containing cocaine, obtaining a positive result from the cobalt thiocyanate chemical screening test and confirmation by GC/MS would be sufficient to identify cocaine. If the analyst cannot prove that cocaine base was present, then FTIR would be the confirmatory choice. If the visual examination indicates that the substance is a plant material which may be marijuana, then the analyst would perform a microscopic examination. If this test is positive for the presence of marijuana, the analyst would then conduct appropriate chemical screening tests to help reach the conclusion of whether marijuana is present or not. Another example is a clear nonviscous liquid which has a strong chemical odor. Performing a Marquis chemical screening test gives a dark red color which indicates opium. The analyst may then run FTIR to confirm that opium is present. Maybe a green oblong tablet is received with markings which indicate that acetaminophen and hydrocodone are present. The analyst could then run a GC/MS to identify both of these substances.

The second scenario would be that a visual examination does not give the analyst an indication as to what substance(s) may be present. If there is sufficient sample available, the analyst must select tests which will narrow the possibilities to one final conclusion. Again generally speaking, chemical screening tests for the most commonly encountered substances are a good place to start because they are quick and simple to perform. After this the analyst could perform UVNIS spectroscopy as a general screening test. One advantage of UVNIS is that it is nondestructive and the sample

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MODULE 17

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Another recovered for further testing. Next would come FTIR spectroscopy which is also non-destructive to the sample. Finally would come GC/MS. It is hoped that the previous tests have given some indication of what substance(s) are present in any before running a GC/MS. If not, there are methods available on the GC/MS for general testing, but they tend to be time consuming and should be used as a last resort.

Selection of microcrystalline testing or TLC for a general unknown is not a good idea because the analyst needs to have an idea of what substance may be present to conduct these tests.

The main scenario is when the sample is a residue with limited sample available for testing. In this case, starting with a general method on the GC/MS may be the best choice as very small amounts of sample can be used. A positive result on the GC/MS will allow the analyst to select an appropriate second test for the substance(s) indicated.

This is when microcrystalline or TLC may be the best choice if they both can be successfully conducted with minimum sample.  
regarding casework

The Trainer will review appropriate sections of the text (see syllabus).

analysis and examination documentation (see Syllabus)

## PRACTICAL EXERCISES

. The trainee will have the opportunity to apply various analytical schemes to the identification of unknown substances by processing casework monitored by the Trainer or designee.

. The trainee will have additional opportunities to apply various analytical schemes to the identification of unknown substances by processing competency samples during the Evaluation of training.

#### DOCUMENTATION ~ ~

The Trainer will review completed case files with the trainee to emphasize the proper format for examination documentation.

Effective date 08-08-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 18

Controlled Substances Training Guide Version 2009

Subject: Reporting of Results, Page 1 of 2

OBJECTIVE

To familiarize the trainee with the proper format for generating reports based on the results of analysis for controlled substance cases.

To familiarize the trainee with the OLO (On-Line Offense) computer system.

DISCUSSION

A lab report is to be generated for all analytical work performed on evidence by the Controlled Substances Section. This report will contain the conclusions and opinions that address the purpose for which the analytical work was undertaken.

Currently, reports of analysis are entered electronically into the Houston Police Department's On-Line Offense (OLO) System. Once an Incident Number has been generated, the officer will enter case related information into the OLO system. The analyst is then able to enter reports as supplied under the same Incident Number. Once entered, the report is printed, signed and dated by the analyst. Only the signed, printed copy of the report that has completed both technical and administrative review will be considered the finalized, official report which is to be maintained as part of the case record.

Each report of analysis for controlled substance evidence should (at a minimum) include the following:

1. header including the unique case identifier.
2. signed.
3. signature of the analyst accepting responsibility for

4. footer including the analytical findings.

The terminology used in reporting the identification of most controlled substances, dangerous drugs, or other substances of interest is determined by the definitions and statutes in Chapters 481-Q-85 of the Texas Health and Safety Code. For some substances only an identification is required. For other substances additional information such as the amount, the isomer form, or the concentration are necessary to meet the statutory requirements for prosecution. The current versions of these statutes should be familiar to an analyst to be sure that all necessary information is available in the final report.

One case may contain evidence from multiple suspects, different locations, or associated with different charges. The report of analysis should be clear as to which items of evidence are associated with these situations. Examples using current reporting guidelines include the following:

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## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 18

### Controlled Substances Training Guide Version 2009

Subject: Reporting of Results Page 2 of 2

NUMEROUS TABLETS 21 ~:2 GRAMS CONTAIN VALPROIC ACID (DELIVERED)  
BAG WITH POWDER 0.2 GRAMS CONTAINS COCAINE (SUSPECT BROWN)  
CIGARETTE 0.01 QUANTITIES MARIJUANA {LIVING ROOM TABLE}  
11 VIALS WITH LIQUID 4.0~ GRAMS CONTAINS PHENCYCLIDINE

(POSSESS 101V SUSPECT JONES)

It is also possible for one case to have multiple reports generated depending upon the circumstances. These reports should be clearly written to reflect the evidence and/or suspects to which they refer without the possibility of confusion.

In addition to using OLO to report the results of analysis, current evidence handling guidelines require that transfers of evidence outside of the Laboratory be documented in OLO (with the exception of evidence released for court). These "reports" should still include (at a minimum) an appropriate header including the unique case identifier.

Examples of statements to document transfers of evidence include the following:

ON 09-31-09 ALL EVIDENCE SUBMITTED IN THIS CASE TRANSFERRED TO IDENTIFICATION OFFICER M. SALDIVAR, PR# 109. THE SAME EVIDENCE WAS RECEIVED BACK FROM OFFICER SALDIVAR ON 10-1-09.

ON 10-01-09 SIX BULLETS, A PLANT SUBSTANCE, GLASS BEAKER, AND A PAPER BAG WERE TRANSFERRED TO IDENTIFICATION OFFICER R. VEROT, PR# 37198. ALL EVIDENCE EXCEPT FOR THE PAPER BAG REMAINED IN LATENT PRINTS.

THE ONE \$7 DOLLAR BILL AND FIVE 2 DOLLAR BILLS IN U.S. CURRENCY RECEIVED IN THIS CASE WILL BE TRANSFERRED TO PROPERTY ROOM.

THE MICROWAVE OVEN RECEIVED IN THIS CASE WILL BE TRANSFERRED TO THE PROPERTY ROOM.

The Trainer will review sections of the CS-50P and the Crime Laboratory Division Quality Assurance Standard Operating Procedures Manual regarding guidelines for reporting results of analysis (see Syllabus Checklist).  
PRACTICAL EXERCISE

The trainee will receive instruction from the Trainer or designee on how to access the OLO computer system to review case related information and for entering supplemental reports.

The trainee will have the opportunity to practice using the OLO computer system to generate reports associated with analysis performed by the trainee and monitored by the Trainer or designee.

### DOCUMENTATION

The Trainer will review completed case files with the trainee to emphasize the proper format for reports of analysis.

Effective date 06-08-09

## MODULE 9

### HOUSTON POLICE DEPARTMENT CRIME LABORATORY

Version 2009

#### Controlled Substances Training Guide

Page 9 of 2

Subject: Case File Documentation

#### OBJECTIVE

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To familiarize the trainee with the documentation which makes up a case file or record.

- To familiarize the trainee with documentation used to track case status and sectional productivity.

#### DISCUSSION

Case records are maintained for every suspected controlled substance case received in the Laboratory. The record may consist of printed documents, electronic data,

of documentation

photographs, or other forms of information. There are three

categories, examination

which make up a complete case record: administrative

i

in one location or in

documentation, and reports. The documents may also be

separated.

multiple locations, but they are all linked by a unique

chain of custody, court orders,

Administrative documents include evidence receipt

documentation includes notations for

phone logs, testimony logs, etc. Examination

process. Reports are generated for all

results of analysis, instrument printouts, preliminary

conclusions and opinions that address

analytical work performed and will contain

information. A case file may be as simple

as a single chair of contemporary work.

As a single chair of contemporary work. B. Analysis each case has separate reports of evidence submitted

Associated with each case will be a Controlled Substances Section analyst fill-out sheet

for that case. For example

Weekly Sheet which lists the cases completed, the number of items and tests

performed, as well as substances identified if any for those cases. The analyst also

documents the weights, volumes, or quantity for



completes a Monthly different subcategory. Some of the case related information such as date received, date completed, number of evidence items, etc., is maintained in an electronic format for easy access when compiling monthly productivity reports.

The Trainer will review appropriate sections of the CS-SOP and the Crime Laboratory Division Quality Assurance and Standard Operating Procedures Manual regarding case documentation and case records (see Syllabus/Checklist).

#### PRACTICAL EXERCISES

The Trainee will receive practice Examination sheets and Weekly sheets to be

reviewed with the Trainer to provide examples of proper documentation of results

and case statistics on these forms.

Effective date 08-08-09

NOUSTON POLICE DEPARTMENT GRIMM LABORATORY MODULE 7  
Controlled Substances Training Guide Version 2009

Subject: Case File Documentation Page 2 of 2

The trainee will have the opportunity to practice completing case file

documentation including Weekly sheets and Monthly sheets associated with analysis performed by the trainee and monitored by the Trainer or designee. The trainee will receive instruction on entering case related information into the electronic Crime Lab Access Database from the Trainer or designee. The trainee will have the opportunity to practice using this database by entering case information associated with analysis performed by the trainee and monitored by the Trainer or designee.

DOCUMENTATION

emphasize

The Trainer will

forms, other

documentation

analysis.

administrative duties

Effective date 06-08-09

## HOI1STOIV POLICE DEPARTMENT CRIME LABORATORY MODULE 20

Controlled Substances Training Guide Version 20~9

Sub~ect: Monitored Analysis Page 1 of 2

OBJECT~VE

To provide the trainee with the opportunity to apply analytical techniques covered in the training program to actual cases under the direct supervision of the Trainer or designee.

### DISCUSSION

An effective way to prepare a trainee to become a qualified analyst is to allow him/her the opportunity to practice applying techniques learned throughout the training program to actual casework or casework simulations. This practice analysis should be directly monitored by the Trainer or designee to provide advice as the work is being performed. The Trainer will select or prepare cases to be processed by trainee. The cases provided to the trainee should be properly identified as submitted for instruction, those previously analyzed by a qualified analyst, or simulations cases. They should also reflect the type of casework which the trainee is expected to process as a qualified analyst.

~

### PRACTICAL EXERCISES

. The trainee will acknowledge receipt cases provided by the Trainer or

designee following standard evidence handling procedures including completing the chain of custody and initialing evidence.

. The trainee will be expected to follow all Laboratory and Sectional procedures regarding evidence handling, analysis, quality assurance, and case file documentation. Trainer or designee will monitor all aspects of casework and will acknowledge initialing all evidence and case documentation. These steps should be noted whether the practice cases are actual submissions or simulations.

. The trainee will generate any necessary reports of analysis with the assistance of

~ the Trainer or designee who will be responsible for signing these reports as the primary analyst. The trainee will complete the Weekly and Monthly sheet documentation and enter all required information into the Crime Laboratory Access Database with the assistance of the Trainer or designee.

. When a case is completed, it will be transferred back to the Trainer or designee following standard evidence handling procedures including completing the chain of custody.

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HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 20

Controlled Substances Training Guide Version 2009

Subject: Monitored Analysis Page 2

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The Trainer or designee will review the completed case files for monitored casework with the trainee to ensure that all documentation has been completed properly. This includes any corrections which need to be made as a result of administrative or technical reviews.

Effective date OB-OS-09

HOUSTON POLICE DEPARTMENT CREME LABORATORY MODULE 2

Controlled Substances Training Guide Version 2009

Subject: Excess Quantity Cases Page 1 of 4

OBJECTIVE

To familiarize the trainee with the term excess quantity as it applies to controlled substance cases.

To familiarize the trainee with statutory requirements for the processing of excess quantity cases.

To familiarize the trainee with guidelines for photographing evidence.

DISCUSSION

Chapter 481.160 of the Texas Health and Safety Code titled "Destruction of Excess Quantities" makes provision for the destruction of property without a court order before the disposition of a case if the agency ensures that (Z) at least five random and representative samples from the total amount of

the property or plant and a sufficient quantity is provided to provide for discovery by parties entitled to discovery; and

(2) photographs are taken that reasonably show the total amount of the property or plant; and

(3) the gross weight or liquid measure of the property or plant is determined, either by actually weighing or measuring the property or plant or by estimating its weight or measurement after making dimensional measurements of the total amount seized.

'

If the property contains a single container of liquid, taking and preserving one representative sample as with Subsection (b).

It is left up to laboratories within the State to determine which cases are to be processed as excess quantity cases as there are not currently any guidelines which specify a size or weight limit. Generally, it is the Controlled Substances Section Lab Manager who makes the determination that a case is to be processed as such and the analyst assigned to the case will be notified of this prior to receiving the case.

It can be seen from the wording above that in addition to following the Section's normal procedures for testing and weighing, that photographs of the entire case submission must be taken and included in the case file. Also, at least five random samples must be retained to represent the whole. The remaining evidence may be destroyed if these conditions are met: Notice that size and weight limits are not specified for the representative samples in the statute. This determination is also left up to the individual laboratories.

Effective date 08-08-09

# H~USTON POLICE DEPARTM~NT CRIME LABORATORY

## MODULE Z~

Version 20U9

Controlled Substances Training Guide

Page 2 of 4

Subject: Excess Quantity Cases

Currently, Section procedures require that excess quantity cases be processed by two

qualified analysts with one acting as the primary analyst responsible for receiving the case on chain of custody documentation and entering the supplement report. The second analyst will assist in all processing and analysis. This way two analysts will be available to testify to the results if necessary.

The Trainer will review appropriate sections of the CS-S~P (see SyNabus/C~ecklisi) for procedures to follow in processing excess quantity cases.

### GUIDELINES FOR PHOTOGRAPHING EXCESS QUANTITY CASES

When photographing excess quantity cases, the following guidelines should be followed:

. All items should be arranged to be clearly visible (i.e., all packages should be can be made more present and accountable) then photographed. So discernable in a photograph by placing a white background behind each individual item. A ruler, yardstick, ruler, coin, size marker, such as standard size 8 1/2 x 1 1/2 inch case identifier and etc., should be present in all photographs. Analysts' initials should be present in all photographs.

. For the purposes of photographing excess quantity cases, the analyst will have the option of requesting the use of the photographer from the Photography Laboratory or utilizing the camera contained in the Controlled Substances Section.

1.

If the Controlled Uses Section camera is used, film will be obtained from the Photography Laboratory.

2.

It is prohibited to photograph no more than one excess quantity case per roll of film.

3.

The completed roll of film should be placed in a Photo Lab submission envelope with a Photo Lab submission form attached, then submitted to the Photo Lab for processing. Ensure that the envelope and form are properly completed. The Photography Laboratory will maintain the case negatives. If the photographs are not received in two days, call the Photography Laboratory.

4.

A videotape may be taken at any time at the discretion of the analyst.

It is acceptable to use a digital camera if the pictures produced are of sufficient clarity. These images should be printed on one of the color printers available in the Laboratory.

Effective date 06-08-09

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 21  
Controlled Substances Training Guide Version 2-09  
Subject: Excess Quantity Cases page 3 of 4

- A~ overview photograph should include the analyst. If all containers cannot be encompassed in one photograph, overlapping photographs should be taken. If the case is processed in parts due to space or time constraints, then each part should be photographed and documented separately to represent the whole.

- Several close-ups, overlapping and from different angles, should be taken of the excess quantity case to ensure proper identification of each item.

- At least one package should be opened to expose contents for the photo.

- Each officer's identifying marks (initials, date, incident number, etc.) if available should be visible in at least one photo.

- The label on all items in bagged, legible

- The analyst

- Close-up of initials, and a size marker

- Items which

- The photograph is 4"x6".

- Inspect the photographs are not acceptable should not be considered for acceptance

- Attach the 31 with the following number (if not in the photograph), the date the photos were taken, and handwritten initials. The photographs are to be placed in the case file.

PRACTICAL EXERCISES

- The trainee may have the opportunity to assist other analysts with the processing of excess quantity cases during the training period.

- After the training period is complete, the newly qualified analyst will process excess quantity cases with a second qualified analyst as per Sectional SOP.

Effective date 06-08-09



## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 21

Controlled Substances Training Guide Version 2009

Subject: Excess Quantity Cases Page 4 of 4

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The Trainer will review completed excess quantity case files if available with the trainee during the training period to emphasize any additional documentation required for these cases.

### CHECKLIST FOR EXCESS QUANTITY CASES

The following is a checklist which can be useful when processing excess quantity cases:

- 

Were the guidelines for photographing excess quantity cases consulted before taking the photographs?

- Are the photographs acceptable as representations of the entire case?

- Were the bulk weights observed and verified by analysts?

- 

Was the tare weighing of the packaging observed by analysts?

- + Was the sampling observed by both analysts?

- Were the analytical results observed by both analysts?

- 

Was the collection and weight of the representative sample observed by both analysts?

- Are all examination documents initialed by both analysts?

- 

Is the supply report complete and both administratively and technically reviewed?

Effective date OB-08-09

## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 22.1

Controlled Substances Training Guide Version 2009

Subject: Trainee Evaluation - Competency Samples Page 1 of 2

### OBJECTIVE

The purpose of competency sample identification is to provide the trainee with an

opportunity to demonstrate his/her ability to apply analytical techniques and procedures to the identification of controlled substances, dangerous drugs, and other chemical substances.

### TESTING FORMAT

The trainee will be provided with a total of 25 powder or liquid samples the identity of which is unknown to the trainee. They will consist of standards or casework samples which have been fully characterized before being given to the trainee. The trainee may use any of the analytical techniques available in this Section discussed during the training period to identify the 25 unknown samples. The trainee is free to use any training materials, notes, or reference literature available to assist in identifying the samples. If the trainee is unable to successfully complete the identification of all 25 samples, he/she will be referred to the Laboratory Director.

### ANALYSIS

#### AND DOCUMENTATION FOR CONTROLLED SUBSTANCE SAMPLES

•

All samples will be documented on examination sheet:

Document one sample per examination sheet.

Label appropriately at the top of column with the sample number and brief description (e.g. "white crystalline liquid")

Document all tests only on the examination sheet.

Attach appropriate analytical documentation in descending order as listed on the examination sheet for each sample.

Ensure that extra and documentations have correct sample number, date and handwritten initials.

Ensure that any necessary dates are documented with the appropriate observations.

Do not weigh the samples.

•

Analytical Sufficiency:

Must have either a GC/MS or an FTIR for each identification.

Must have at least one additional positive test. All negative spot tests are NOT acceptable as the additional test. A retention time on the GC/MS that matches the retention time of a standard is NOT acceptable as the additional test.

Effective date 08-05-05

HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 22.1  
Controlled Substances Training Guide Version 2Q  
Subject: Trainee Evaluation - Competency Samples Page 2 of 2

All samples must be identified. No Controlled Substance is not an acceptable identification. There will be something in every sample to identify even if it is not a controlled substance. A few of the samples will have more than one substance to identify.

If a sample contains cocaine, methamphetamine, or heroin, a percentage of purity must be determined {unless otherwise noted by the Trainer}.

Appropriate quality checks will be documented for any chemical screening tests that are not frequently used (e.g. Koppanyi or FeCl<sub>3</sub>).

For the 25 collective competency samples:

Competency must be demonstrated on all available analytical instrumentation by using each instrument at least once. There will be NO assistance provided in using procedures or use of the instruments. If you have a problem with the Trainer, do not ask other analysts for assistance.

•

Guidelines for handling and competency samples:

All trainees should be following a guideline for having only one sample open at a time to prevent possible contamination or deleterious change.

When samples are not being processed, they should be secured in the trainees work area. Handle the lid and do not leave them sitting out while you are absent (e.g., overnight).

All trainees should follow laboratory guidelines regarding documentation {handwritten initials on all paperwork, proper quality check documentation for reagents, titration balances used for purity determinations etc.}.

When the analysis is complete, turn in the samples and the paperwork with all appropriate spectra, documentation, and identification to the Trainer.

Trainees will have a full workweek to complete the 25 competency samples. If the samples are received by the trainee on a Monday morning, they will be due by the end of the day on the following Friday.

It is not advisable to wait until the last minute to turn in paperwork and samples. It is better to complete samples as you go and turn them in accordingly. The Trainer can review the trainee's progress and make suggestions if there are gross errors or omissions (e.g. don't forget to use all available GC/MS). It is acceptable to request a sample back even if you have already turned in the paperwork up to the final due date.

Effective date 08-08-09

## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 22•Z

Controlled Substances Training Guide Version 2009

Subject: Trainee Evaluation - Final Written Examination Page 1 of 1

### OBJECTIVE

The purpose of the final written examination is to provide the trainee with an opportunity to demonstrate technical knowledge related to the analysis of controlled substances, dangerous drugs, and other chemical substances as provided by the Controlled Substances Section Training Program.

### FINAL EXAMINATION STUDY GUIDE

Accreditation:

- By whom is the HPD lab accredited?
- When did the lab first receive accreditation and when does it currently end?
- What are the three types of criteria by which it is measured and what

percentage of each criteria must be met in order to achieve accreditation by

ASCLD-LAB?

- Distinguish the terms accreditation and certification.

Drug Control Policies:

- Understand the basis of the Federal Controlled Substances Act and the

Scheduling of substances Federal Level (i.e. How many schedules are there and what are the criteria for placing a particular substance into one of the schedules?)

• Be able to define the terms "Controlled Substance", "Dangerous Drug", "Simulated or Imitated Substance", "Adulterants and Diluents", and "Marihuana" based on the current Texas Drug Laws.

• Understand the difference between Controlled Substances, Dangerous Drugs,

and Over-the-counter substances in Texas.

- Be able to identify the Penalty Group for various substances (see table).

Drug Classifications, Effects, Structures, and Isomers:

- Understand the terms Narcotic, Depressant, Stimulant, and Hallucinogen in

relation to the effect of a substance on the body and be able to identify the effect

on the body of various substances (see table).

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- Understand the chemical structure relationships between substances that have similar effects on the body e.g. recognize similarities in the structures of natural opiates, benzodiazepines (pam-lams), amphetamines (phenethylamines), steroids, etc.

- Be able to match the chemical name for various substances with the provided structures.

- Understand the similarities and differences in the free base vs. salt forms of cocaine and methamphetamine. What is "crack", how is it made, and how is it abused? What is "ice", how is it made, and how is it abused?

- Understand the term opium, its source, how it is abused, 5 principle alkaloids present and their relative abundances. Understand the narcotic as well as be able to identify various narcotics as naturally-occuring synthetic, or synthetic.

- Understand the terms structural isomers, geometric isomers, diastereomers, optical isomers, and enantiomers. Be able to give examples of each.

Spot Tests & Chemical Screening Tests

- Understand the reagent guaiacum system used by the Controlled

Substances Section. Identify the reagents used for spot tests as frequently used or infrequently used.

- Understand how to perform various spot tests and the expected results for commonly encountered substances (see table).

- Understand the most common Spot Tests as a preliminary identification for substances including the following:

Ferricyanide Secondary amines

Marquis Opiates, Amphetamines, Designers,

Toluene, Aspirin

Van Urk's Selected Local anesthetics, LSD

Cobalt thiocyanate 1 Modification Cocaine, PCP, GBL

Duquenois / Duquenois-Levine Cannabinoids

Ferric Chloride GHB

Janovsky Selected Benzodiazepines

Weber Psilocyn, Psilocybin

Koppanyi Barbiturates

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## HOUSTON POLICE DEPARTMENT CRIM~ LABORATORY MODULE 22.2

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Module Objectives:

- Understand the reagent quality control system used by the Controlled Substances Section. Identify various reagents used for microcrystalline tests as frequently used or infrequently used.

Understand how to perform various microcrystalline tests and the expected results for commonly encountered substances (see table).

UVNIS Instrumentation and Analysis:

- Know the principle components of both single beam and double beam instruments.
- Know the general scanning range and the acceptable range for a UVNIS spectrum.
- Be able to define the terms "chromophore", "hemic shift", "bathochromic shift", "hyperchromic shift", and "hypochromic".
- Understand the principles of ultraviolet energy absorption and which types of excitations are respectively routinely observed spectra.
- Understand why spectra for narcotics may look similar and why some substances do not produce a spectrum.

Be able to match provided spectra to a list of possible substances

including the following:

Acetaminophen

Alprazolam

Amphetamine, Methamphetamine

Benzocaine and Procaine

Barbiturates (Amobarbital, Secobarbital, Phenobarbital, etc.)

Caffeine

Carisoprodol

Cocaine

Codeine

Designers (MDA, MDMA, MDE)

Di2~zepam

Heroin

Ketamine

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L~docaine

LSD

Methadone

Phencyclidine

Know the eqt~ation to relate Absorbance to Transmission and the Beer-Lambert ~.av

$$A = \log (1/T) = abc$$

where A is a~sorbance and T is transmittance

a is a constant specific to t~e substance of ir~terest at a particular waveleng#h

b is the pathieng#tt ir~ centimeters

c is the concentration in mg 1 ml at HPD

Be able to calculate % purity of a sample from a providsd~VNES s~ectn~m.

Be able to calculate the E value for a peak from a pro~~U~t/IS spectrum.

•

Be able to convert amounts and % purity be s~ free base forms of provided substances. ,

Separat~ons 1 Extractions:

•

U~derstand pH measurement of aci r~ asic solutions and how such solutions are prepared.

•

Understand the terms "mis ' e "immiscible" and apply tl~em #o combinations of solven~

•

Know t~e g~neral ~cheme and apply it to the se~aration of weak actdic, strong i n al, and basic s~bstances. .

• Be able t p~ain e principle and use of a Conway extraction.

. Be able to perform various separations and extractions for competency samples including CHC13 washes, bicarb washes, KMnO4 extr, and A/B extr.

FTIR Instrumentatfon and Analysis:

•

Know the general scanning range for IR spectra and the units commonly used for measurement of energy.

U~derstand the principles of IR energy absorption by molecules and the types of iransitions that take place including vibrativnal stretching (symme#ric and asymmetric), vibrational bending (scissoring, twisting, wagging, and rocking), and molec~lar rota#ions.

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o  
Be able to name and define the three types of IR ~sar~tien ba~cls i!~cl;~diny  
----- ~ ~~~ ~~~  
fundamental, overtar~e, and combination bands.

- Know the ~rir~ciple components of both dispersive and Fourier transform instruments.
- Be familiar with the two main advantages of FTIR spectrometers over dispersive instruments ("throughput" and the "multi~lex" ad~antages).
- + Know the two uses for a He-Ne lase~ in an FTIR instrument.
- Know the meaning of the terms constructi~e and interference, and refraction.
- Be #amii~ar with the basic principles of attenuated ;e (ATR} spectroscopy including the types of crystals co~ Se, Ge, Diamond) and be able to de#ine the term evaRg~g~s~

- Know the significance of air and water in I
- Be ab~e to identify the functional gr ~ far various infrared absorption bands inclucfiRg the followir~g:

N-H stretch {3500 cr n -'

CO2 stretch (2300-2~ )~

-11

C-H stre#ch {29fi ~~,

O-H stretch (32 -3+ i cm~~,

C=O stretch ~ Ocmr )

~

- 

Be able to discuss significance of the "fingerprint" region in an IR spectrum.

- 

Be able to discuss the variation observed in the  $C=O$  stretching region of the FTIR spectra for cocaine base and cocaine HCL.

Be able to match provided FTIR spectra to a list of possible substances including the following:

Amoxicillin

Ampicillin

Carisoprodol

Cocaine base

Cocaine HCL

GBL

GHB

Write a note

Heroin HCl ----~  
 Methamphetamine HCl  
 Toluen~

#### GCIMS Instrumentation and Analysis:

- Know the principle components in a GC/MS system and the purpose of each (GC: injector, column in a temperature regufated oven, source of camer gas ; MS: ion saurce, mass separator (quadrapole or ion trap), electron mt~ltiplier detector ; Data anafysis system)

- Know which factars can be adjusted to increase separation efficiency in a GC system. ~

- Be able to define retention time. Know the effect that n radation~ will ha~e on separation efficiency and retention time K ect that changing various GC parameters such as temper~ture, c mn coating thickness, and co~umn length will have on retention time.

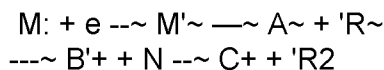
- Be able #o predict the mos# iikely elt~t~on • r a mixture of substances using a non-polar GC column.

- Know the carrier gas used in tt~e L M5 systems and know the advantages of ~arious gases~a o fficiency and safety.

- Know the compound m nly used to calibrate (tune) the mass axis of the mass s~ectromete

- Be able to ex i hy MSD system should be run under a vacuum.

- Be able to ~' cus lectron impact ivnization in a MS system and how compound fragmentatia rs. Know the standard energy of electrons produced by the filament in an MSD source. Be able to identify the species which would be de#ectsd in an electron impact mass spectrometer from the fol~owing equation:



Know the units commonly used for Totaf Ion Chromatograms and Mass Spectra. Be abie to discuss variations in the fragmenta#ion patterns of aliphatic vs. aromatic compouRds.

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Be able to fdentify the Base peak and the ~arent peak from a provid~d m~ss spectra.

•

ge able to match provEded GC/MS spectra to a list of possible substances including the following:

Acetamir~ophen

Alprazolam

Amphe#ami~e

Carisoprodol

Cinnamoylcocaine

Cocaine

Codeine

Heroin ~

Hydrocodo~e

MDA

MDMA

Met~amphetamine

Monoacetylmorphine

Phencyclidine

Procaine

Promethazine

General Instrumentation and Ana

• Which anafytical techn for salt 1 base form determination?

. Which instrn.rme is preferred for identification of barbiturates?

. Which instrum hnique is preferred for identification of antibiotics?

•

Whic~ analy niques are considered destructive and which are non-destructi~e?

Marihuana A~alysis:

Be able #o define the teRn "Mari~uana" based on the c~rrent Texas Drug Laws.

Be able to expiain the control status of °marihuana" and "THC" based on the current Texas Drug Laws.

Which parts of the marihuana plant a~e not controlled?

W~at is the term used to describe the unique chemicals found in mari~uana?

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o What is the main psychoactive ingredient found in marijuana (full name, not just abbreviation)?

- Know that marijuana / THC is generally considered to be an Hallucinogen.

~  
Be able to match the name for various chemicals found in marijuana with the provided structures.

- Be able to identify the various parts from the cross section of a bract from the fruiting cannabis plant.

- Be able to provide the complete botanical taxonomy of the marijuana plant.

+  
Be able to explain the proper way to conduct the chemical reagent tests for the presence of marijuana / THC. Know the ingredients in reagents used for these tests. ~^~

Testing Format ~

The examination consists of short answer, and multiple choice questions. Each question will have the point value clearly stated. There will be an opportunity to obtain extra credit during the exam. The trainee will be provided with a private, quiet area in which to take the exam. Scratch paper and a calculator are permitted. The trainee will need to answer 90% or correctly to pass and proceed with the training program. If the trainee fails the exam, he/she will be referred to the Laboratory Director.

Point Breakdown:

Total Points = 332 mg = 90% = 298 points Extra Credit = 33 points

Breakdown of

Accreditation: ~3 points

Drug Control Policies: 30 points + 3 bonus

Drug Classifications and Effects: 40 points + 4 bonus

Structures and Isomers: 56 points + 10 bonus

Spot Tests: 17 points

Microcrystalline: 10 points

UVNIR Instrumentation and Analysis: 45 points + 2 bonus

Separations / Extractions: 12 points

FTIR Instrumentation and Analysis: 40 points + 4 bonus

GC/MS Instrumentation and Analysis: 38 points + 3 bonus.

General Instrumentation and Analysis: 4 points

Marijuana: 30 points + 11 bonus

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Substance Contro#Status--Effect 1 Use Spot Tests Microcrystalline

Penal Grou

Cocaine base/NCI 1 Stirn Co SCN 2 A. Au Pt

GHB 1 De FeCl3

GBL 1 De Co SCN 2

LSD 1-A HaA Van Urk's

Heroin 1 Narc 5S Mar uis

Qx codone 1 Narc SS Mar uis

MDA 2 HaEI Mar uis

MDMA 2 Hall Ferricyanide

Mar

Meth 1 Stim Ferric nt Hanging Drpp

Acidic Au, Pt

Hydrocodone 1 1,3 Narc (SS) is

Dih drocodeinone

Am hetamine 2 Stim ar uis

H dromo hone 1 Narc S Mar uis

Al razofam 3

pCp 1 I Co SCIV 2 A. Au, KMnQ4

Codeine 1 3 4 Mar uis

Mo hine 7 arc N Mar uis

Deri~atives of 3 Dep Kappanyi

Barbituric acid

Procaine Van Urk's

Benzocai~e Van Urk's

Toluene ~e Mar uis

Pseudaephedrin Precursor

Decon estant

Carisoprodo! DD Muscle

Reiaxant

Diaze am 3 Bersz De Jano~sk

Psilocin 1 P5iloc in 2 Half Weber

Fentan 7 Narc S Mar uis

Dextro ro ox hene 3 Narc S

Guaifenesin Ex ectorant Mar ~is

Dextromethorphan Cough Marquis

Su resant

Phen e hrine Decon estant Ma ufs

. AcetamEno hen Anaf esic

Chlor heniramine Ant[hfstarnlne

As trin ~ Anal esic Mar uis

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S~~bst?~Ce Contr~~Stat~s Eff~ci / ~~~eSpot i ests1Vlicrocrystafiine

Pe~al Grou

Flunitraze am 1 De Janovsk

Pe ote 3 hiall Mar uis  
Mescaline 2 Hafl Mar uis  
Ketamine 1 Hall

Meth enidate 3 Stim Ferric anide  
Methadone 1 Narc S  
Testosterone 3 A5  
Cannabinoids 1 THC 2 Hall Duquenois  
Sct~edule 1  
Marihuana (own penalty group)Hall Duque '  
Schedule 1

~

Abbreviations Used in Tat~le:  
Narc (N) — Natural Opiate Narcotic HaEl —  
Narc (SS} — Semisynthetic Narcotic  
Narc (S) -- Synthetic Narcotic C depressar  
AVC — Abusable Volatile Chemical D gerous Drug  
Benz — Benzodiazepine — Anabolic SteroEd  
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TEST~MONY READfNG LIST

(to be initialed when completed)

~) R. Saferstein editor, Forensic Science Handbook, Vol. 1, 2"d Ed., 2002.

Ch. 1—"Legal Aspects of Forensic Science"

2) J.E. Horsley, Testif in in Court, 4972.

3} D. Poynter, The Ex ert Witness Handbaok, ~ 987.

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## HOUSTON POLICE DEPARTMENT CRIME LABORATORY MODULE 22•3

Controlled Substances: Training Guide Version 2009

Subject: Trainee Evaluation - Testimony and Mock Trial Page 2 of 14

### OBJECTIVE

To familiarize the trainee with courtroom proceedings in a criminal case.

•

To familiarize the trainee with proper methods of presenting expert testimony during trial.

### DISCUSSION

"Expert Testimony" is defined as "... the opinion of a witness who has special knowledge, wisdom, skill, or information regarding a subject of inquiry, acquired by study, investigation, observation, practice or experience, and unlikely to be possessed by the ordinary layman or inexperienced person incapable of understanding the subject without the aid of some person having such special knowledge." Robert L. Donigan and Edward C. Fisher, The Evidence Handbook, Traffic Institute University, p. 147. ) ~ ~ ~

As an expert witness, a person is permitted to respond to questions more than an ordinary witness. The analyst's opinions in their field of expertise, provided those opinions are drawn from facts and observations. It is wise, however, to keep in mind the limits of a person's experience. One should not allow them to be led too far afield into areas outside their personal expertise (i.e., drug effects, toxicology, street doses, street logical questions, etc.).

How does a person's training, and experience in drug identification witness's expertise. Only a presiding judge can rule on the qualifications. The prosecuting attorney will examine the witness. The defense attorney must decide if the qualifications become all important.

The first thing to consider is the matter of appearance. Personal clothing should be neat. Coats and ties for male analysts are a necessity. Women should, likewise, dress in a conservative, business like fashion. The jury or potential jurors attach significant importance to the attire and appearance of the forensic expert. Another facet of appearance is the personal demeanor while on the witness stand. One should be careful not to rock or swivel unnecessarily and to avoid any personal idiosyncrasies and/or nervous habits, such as playing with the case file or parts of clothing, nervous twitches, etc.

The manner of answering questions is also important. Answers should be delivered in a moderately pitched, calm voice, slowly and loudly enough to be understood, yet not so slowly as to seem hesitant. Answers should be directed to the jury as much as possible. Good eye contact should always be maintained. In the final analysis, the jury will decide the outcome of the case on its merits. If called upon to explain a scientific

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## HvU~~OIV pGLIGE DEPARTMENT CRIME LABORATORY MODUL~ 22.3

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Se~biect: Trainee E~efuation - Testirngnv and Mock TriaE Paae 3 af ~~4  
concep#, such as IR or TLC, t~e expert witness should do so in clear layman's  
language.

The main purpose of #estimony by a forensic ar~alyst is to con~Ey to the jury or judge the  
~eracity and com~leteness o~ the testing proc~dures, not to confuse them. It is vital that  
the analyst formulate in their own mind a clear, concise explanation of tt~ese techniques,  
whicF~ can be r~adily understood by ~aymen of a~erage fntelligence. In other words, any  
appearance of fecturing tt~e jury should be avoided. When answering questions posed  
by the attorneys or the court, one s~ould always be courteous. The use of "Your Hono~"  
when referring to ti~e judge or "sir" or "ma'am" when answering the attorneys is  
desirable and appropria#e.

Response to cross examination should be polite, firm, and over expansive or  
arg~mentat~e. One should never argue or be rtasty, sarcasti~ r~fling witf~ defense  
counsel. If the answer to a question is unknown, one should~ ~sitate to reply so.

One should fully ~nderstand any questions posed by f a witness is not  
certain, it is proper to ask for a repetition f~i {Would you please  
repeat/rephrase the questian?}. If an a~swer to a q uires some thought, or~e  
should not be afraid to hesitate before answering instat~ces, it is important to  
hesitate for a# least half a second be~ore ans~r g in~ to permit the prosecutor to  
object if the analyst feels it necessary. ~,, \_

During vigorous cross-examination an ~pe~xchanges w#th defense counsel, one  
should not hurry answers or raise one oi Instead, a witr~ess should make an effort  
to remain calm. Frequently, a que be asked for which counsel demands a yes  
or no answer, i.e., "Is it fair to sa or i't no# so) that TLC is not s~ec~c for LSD?". In  
such a case, it may be nece ate that this question cannot be answered by a  
simple yes or no answer. 'u e w~ll usually allow an explanation~ to the answer.  
The purpose of an e p ~ is to eniighten the court on matters within the field of  
expsrtise. Ir~ order t 's rg this responsibility, questions must be answered fully. If  
yes or no fails achi ese objecti~es, or~e sl~ould nat hesitate to iRdicate to the  
court t~at the ~u~n e not be answered wi#h a yes or no response.

During both direct and cross-examination, there are a number of thir~gs to remembe~.  
Again, one should a~ways be courteous when either side raises an objection during  
testimony. TY~e witness should imm~diately stop and proceed on~y upon direction and  
instructions of the co~rt.

Certain phrases should not be used such as:

'To teli you the truth, . . . .

"Frankly, . . . .

"To be honest, . . . .

"I think . . . .

"! befieve . . . .

T~ese a~d any other casuaf, flip~ant terms and phrases should be a~oided.

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During the direct examination, the prosecutor will establish the qualifications of the witness. Once the witness has been qualified, the prosecutor will attempt to introduce the evidence and elicit the results of analysis. By the time the analyst is called to the stand, the agents or police officers in the case normally will have already testified as to the circumstances under which the drug was purchased, transferred, or seized, and what preparations they made to deliver the evidence to the laboratory. The first question, will usually establish the chain of custody, -- how the exhibit came into the possession of the analyst. "How do you know this is the exhibit you worked on?" The importance of labeling and initialing the evidence envelope or other container is clearly seen at this stage of the trial. The analyst may be asked to break open the seals and withdraw the evidence or the evidence may have been previously opened by the agents. The next question will ordinarily concern the analysis, -- "What did you do upon receipt?" "What did you find?", etc. Often, the prosecutor will discuss how the analysis was performed, preferring to leave that questioning for the defense.

It is a good rule, in most instances, to answer only the proposed and nothing more, unless called upon to give explanations. One should not volunteer information. In addition to the role as expert witness, an analyst may serve as advisor to the prosecuting attorney. This assistance can take various forms. In most instances, the prosecuting attorney will wish to hold the analyst in reserve. Although the analyst appears only occasionally at these conferences, they can be extremely helpful, particularly if the prosecutor is inexperienced or not acquainted with the witness. In such cases, it is frequently helpful for an analyst to present a resume/qualification sheet and list of questions to serve as a guide for the direct examination. In many instances, since the forensic analyst's testimony is relatively straightforward, the pretrial conference may consist of a fairly short conversation with the prosecutor.

Occasionally, after testimony, an analyst may become aware of flaws or additional facts that were overlooked by the prosecutor or that were brought up in cross-examination and require further clarification. Any such instances should be brought to the prosecutor's attention in a discreet, diplomatic manner as soon as the situation allows. In most cases, a drug trial will never even begin unless an analyst has reported the presence of a controlled substance. The impartiality of the analyst begins in the laboratory. Above all, the analyst must maintain a completely open mind as to the nature of every exhibit. It is possible that a person has been falsely accused. No controlled substance is present. In such an instance, it is the obligation of the analyst to report this fact and no court action should ensue. Nothing can destroy the credibility of an expert witness as rapidly as an impression on the part of the jury that the witness has a strong interest in the outcome of the trial.

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### TESTIMONY MONITORING

The Trainer will review the appropriate section of the Crime Laboratory Division Quality Assurance and Standard Operating Procedures Manual regarding courtroom testimony and monitoring.

### PRACTICAL EXERCISES

- 

Throughout the training period, the trainee should have the opportunity to observe qualified analysts testify in court.

- 

The trainee should have the opportunity to view tapes of mock trials for previous trainees. ~

- 

The trainee opportunity to testify if available.

- 

The trainee should practice answering cross examination questions in preparation for a mock trial.

### DEFINITIONS

The following are some general concepts which may be useful to a controlled substance analyst. These are in addition definitions listed in Chapters 481-485 of the Texas Health and Safety Code

- 

Addiction - Drug addiction is a state of periodic or chronic intoxication produced by the repeated use of a drug. Its characteristics include:

- . An overwhelming desire or need (compulsion) to continue taking the drug and to obtain it by any means;

- . A tendency to increase the dose;

- . A physical (psychological) and generally a physical dependence on the

effects of the drug;

- . An effect detrimental to the individual and to society.

- 

Alkaloid - Group of basic, nitrogenous plant products which have marked physiological action. The majority of these are complex heterocyclic compounds.

- . Analgesic - Insensitivity to pain without loss of consciousness; pain killer.

- . Anesthetic - Causes loss of sensation with or without loss of consciousness.

- . Anorectic - Causes loss of appetite.

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By-Products - Compounds found in acidic media that are formed from the reaction of a chemical reaction or an extract.

•

Central Nervous System (CNS) - The brain and spinal cord.

- Central Nervous System (CNS) Depressant — A substance that lowers the heart rate, respiration, and blood pressure. Medical uses include the treatment of anxiety, tension, and high blood pressure.

- Central Nervous System (CNS) Stimulant — A substance that increases the heart rate, respiration, and blood pressure. Medical uses include the treatment of mild depressive states, overweight and narcolepsy, a disease characterized by an almost overwhelming desire to sleep.

•

Criministics - The science which involves the physical sciences (e.g., analysis, physics, biology) of the crime.

•

Dependence - Drug dependence is psychological or physical dependence or both, which results from the continuous use of a drug. —

•

Felony - A grave crime declared by the common law or by statute regardless of the punishment attached.

•

Forensic Science - Forensic is a broad term denoting the application of medical, social, behavior sciences to the administration of justice.

•

Habituation administration of a drug, which includes

- a desire (but not a compulsion) to continue taking the drug for the sense of

- improvement in being that it engenders;

- little or no tendency to increase the dose;

- some degree of psychic dependence on the effect of the drug but absence

of physical dependence and, hence, no abstinence syndrome;

- a detrimental effect, if any, primarily on the individual.

Simplified, habituation is the psychological desire to repeat the use of a drug intermittently or continuously because of emotional reasons.

Hallucinogen - Both natural and synthetic hallucinogens are substances that distort the perception of objective reality. They induce a state of excitation of the central nervous system. A person using hallucinogens will be disoriented, have delusions, and hallucinations,



~~ective date 06-OS-09

- Hypnotic - An agent that induces sleep.
- Impeach - To discredit a witness.
- Local Anesthetic - Causes a numbing effect.  
~ Misdemeanor - A crime less serious than a felony. For possession Penalty  
Gfvup 3 and 4 substances weigh~ng less t#~an 2\$ grams fncuding adul#erants  
and dilutents. For ~ossession mar~huana weighing less than 4 ounces. For  
possession dangerous drugs witho~t a prescription.
- Narcotic - Something that soothes, relieves or lulls; dnag that in moderate doses  
dulls the senses, relieves pain, and induces profound ep but in excessive  
doses causes st~apor, coma, or oon~ulsions (dictionary d n ). This term in its  
medical meani~g refers to opi~m a~d opium derivati s hetic s~bsti#utes.  
They are t~e most effecti~e agents for the reli of n are centra! nervous  
system (CNS) depressants.
- Opiate - A substa~ce that ~as an add' ir~g or addiction-sustaining  
liability similar to morphine or is cap of onversion into a drug having  
addiction-forming or addict~an-sust " ia . The term incle~des its racemic  
a~d levoro#atory forms. The t~ s not include, uness speci~ically  
designated as controlled under '0 81.038, the dextrorotatory isomer of  
dextromethorphan (from Tex tro d Substances Act}).  
. Over rufed - Whe~ an ' n over n.ile~ you can answer tf~e question~.  
. Perjury - Act of wil ring or #estifying falsely.  
. Physical epe e - An adaptive state caused by repeated drug use tY~at

reveals i f by developmen# of intense physical symptoms (withdrawal  
syr~drome) se of the drug is stop~ed.

- Potentiavn - Occurs when the combined act~on af two or more drUgs fs greater  
than the sum of the effects of each dn.rg talcen afone. Potentiation can be very  
useful in certain medical procedures. For example, physicians can induce and  
maintain a s~ecific degree of anesthesia by using another drug to potentiate the  
primary anesthetic agent. Potentiation may also be dangerous. Far example,  
barbiturates arid many tranquilizers potentiake the depressant effects of aloohol.  
Psychological Dependence - An attachment to drug use which arises from a  
dn.ig's\_ability to satisfy some emotional or personality need of an individual. TF~is  
attachment does not require a physical dependence, although physical  
dependence may seem to reinfarce psychological depende~ce.

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~ Sedative - An agent that quiets or calms the patient.

- 

Sustained - When an objection is sustained, you cannot answer the question.

~

Tolerance - With many drugs, a person must keep increasing the dosage to maintain the same effect.

- Voir Dire - Preliminary examination of witness (e.g., criminalist) in order to determine qualifications and competency, or to examine in depth the chain of custody of the evidence.

The following are abbreviated definitions that could be useful when testifying about certain testing procedures. ~

- 

Pharmaceutical Identification - Comparison of pills or tablets, capsules, or containers with recognized trademarks to make presumptive identifications as to the contents and of pharmaceutically manufactured products.

- 

Spot Tests - Series of presumptive chemical tests which indicate what type of compounds might be present.

- 

Microcrystalline Tests - An unknown sample is dissolved in a reagent and viewed under the microscope - formation of characteristic crystals.

- 

Thin Layer Chromatography - Comparison of adsorption rates of a known and an unknown sample.

quant system on a silica gel surface.

~~~~

-

Gas Chromatograph - An instrument which separates and identifies the components of a mixture based upon their retention times on a separating column. ~

-

Gas Chromatograph Quantitation - A quantitation based on the comparison of the areas under the peaks for an unknown sample and a standard.

- Ultraviolet-Visible Spectrophotometer (UVVIS) - An instrument which identifies a sample based on its light absorption pattern. Ultraviolet light at different wavelengths passes through the sample and a graph is produced!

showing where the light is absorbed. Starting with a known sample weight and a known solvent volume, the percentage of purity can be determined.

- Gas Chromatograph/Mass Spectrometer {GC/MS} - An instrumental technique which separates and specifically identifies the components of a mixture based upon their masses.

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o Fourier Transform infrared (FTIR) - An instrumental technique which identifies substances based upon their unique infrared light absorption patterns.

•

By-Products - Compounds found in addition to the compound of interest after a chemical reaction or an extraction.

•

Instrument - A device that uses scientific principles to make accurate, precise, and repeatable measurements.

• Machine - A device that converts energy into motion or motion into energy.

WHEN TESTIFYING IN COURT

The following are suggestions to remember when testifying •

•

Look at the jury if an answer is longer than a

•

Speak loudly, clearly, and slowly!

•

Sit up straight, don't lean forward. Use the chair.

•

Focus on the question. Answer a complete question has been asked (provided there is no sustained objection). If you don't understand the question, then say so. If you don't know the answer to the question, say so.

•

Avoid technical jargon - simple terms.

• If appropriate, estimate when giving weights or percentages.

• If the judge asks you a question, answer the question,

S

ANSWER--don't say anything.

•

Don't volunteer information. Answer only the question you are asked.

•

Try to remain calm and do not show outward signs of becoming upset. This

applies to both direct and cross examination.

• Don't change demeanor or appearance when you are passed for cross examination.

• Be cautious of defense attorneys that are too friendly or helpful.

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Remember that you are testifying to your opinion as a expert witness of controlled substances. Do not testify or answer questions outside of your training and experience.

Do not give the impression of being for the prosecution and against the defense (or vice versa). Just testify to facts to the best of your knowledge whomever is asking the questions.

COURT QUESTIONS

Direct Examination

Please state your name.

What is your occupation and by whom are you employed?

How long have you worked there?

What is your title? Exactly what is a Criminalist?

What education and training do you have in order to work as a Criminalist for the HPD Crime Lab?

Do you belong to any professional organizations?

Have you published any articles?

Have you testified as an expert before?

Are you certified?

Is the Crime Lab accredited? By whom?

What are your duties at the Crime Lab?

Is it possible to take an unknown substance and determine its identity?

Have you done this on few or many occasions? Approximately how many?

How do you do that?

Let me call your attention to State's Exhibit #1 (evidence envelope) and ask if you can

identify it. What is it? How can you identify it?

Does the inventory on the outside of the envelope match with the contents?

What is a submission form? Who prepares it? Does it always match what has been written on the evidence envelope?

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When was the first time you saw State's Exhibit #1?

Under what condition was it when you received it?

From whom did you receive State's Exhibit #1? Where did they receive it?

Are those business records you are testifying from? Are you the custodian of the records for the Houston Police Department Crime Laboratory? Are the entries on those records made at or about the time of the event by someone with personal knowledge of the event?

How are samples submitted to the HPD Crime Lab? What is the Lock Box? Where is it located? Who has access to the Lock Box?

What did you do with State's Exhibit #1 after you received it?

Let me call your attention to State's Exhibit #2 (the envelope) and ask if you can identify it. What is it? How can you identify it?

Did you have an opportunity to perform an analysis of State's Exhibit #2?

Examples of Analysis Questions

What tests did you perform?

You said you performed a series of tests/screening tests. What are they?

What were your results from screening tests in this case?

Is it possible for different substances to give the same result with a spot test?

You said you ran an ultraviolet test. Please explain how you did that. What were your results for the UV test? Is this a specific test? What other substances can give the same result?

What is a microcrystalline test? What were your results from the microcrystalline test?

Is it possible for other compounds to give the same crystals?

Explain the GC test. What were your results for the GC test? Is this a specific test?

How does the GC give you a percent purity?

Explain thin layer chromatography. What were your results for the TLC test? Is this a specific test? What other substances might give you the same result?

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Explair~ FTiR. _What wereyouur_results for_thQ FT~R? !G this ~ sp~~ifc :~st? ',Nha~ ath~;
substances might gi~e yoU the same rssu~t?

Explafn GCIMS. What were your results for the GC/MS test? Is this a speci~c test?

What other substances might give you the same result?

Did you form an opinion as to the identity of State's Exhibit #2? What is that opinion?

Is that a controlled substance in the State of Texas? What is a con#rolled substance?

Did you have an opportunity to weigh the contents of State's Exhibit #2? How muc~ did
it weigh? is that #he aggregate weight including any adulterants or dilutants? What is
an adulteran# or dilutant? Is that a weigF~t that is iess than one ram (whatever range is
appropriate)?

Did you perform a qualitative {quantitative) analysis? What we o esults?

~

W~at are the physical effects of the sUbstance you

Wha# did you do witl~ the evidence when you we it?

When was t~e last time tha# you saw ~ Does it appear to ha~e been

tampered wit~ in any way since the las#

Cross Examination

Do certarn substances have l compositions?

Is it possible for another s have a similar type of reaction or result?

How often are you

Would you know were contaminated?

Did you test for other controlfed subs#ances?

Did you identi~y the adulterants or difutents? Why not?

Do you have personal knowledge of where these dn~gs came from?

Do you have independent recollection of worki~g this case?

How many cases do you open at one time?

How do you secure your evidence?

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How do you ensure that other Bvirig-rg ~r~e~ r~o: cc~^~e ~~~to ~,on~act c~r g~# mixed
wiiH

otFier cases?

Why do you perform so many tests; aren't you sure about what you have?

~s there a margin of error in each test?

Do you work for other agencies other than the HPD?

Are you a State witness? Do you always testify for the State? Are you being paid for your testimony?

Do you know my client? Do you have personal knowledge that this evidence was taken from my client?

Do you know if this evidence was examined for fingerprints? w n't it?

Did you do DNA testing on this evidence? Why I t have been tested for DNA?

- Examples of Marijuana Questions

What tests did you perform?

You said you performed a microscopic examination of the plant substance. What were the results of that examination? some of the features of marijuana that you are looking for under the microscope Are you trained as a botanist? Is this test specific for marijuana? Is there other substance that could give you the same result?

You said you performed cal/spot/screening tests. What were they? What were the results of that test that chemicals are contained in the solutions used for those tests? Did you prepare those solutions? How do you know the solutions are any good? How do you know whether or not the solutions are contaminated? Are these tests specific for marijuana? Are there other substances which could give the same result? Did you form an opinion as to the identity of State's Exhibit #2? What is it? Is marijuana a controlled substance?

How many samples did you take to conduct your tests? Did you perform an analysis of the rest of the substance? How do you know that it is all marijuana?

Did you have an opportunity to weigh the contents of State's Exhibit #2? What was the total weight of the plant substance? Is that a useable quantity? Does that include any adulterants or diluents? Does that weight include seeds? Why didn't you remove the seeds before weighing? How do you know that the seeds are not sterilized? What would be the weight without the seeds? Would it be less?

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MOCK TRIAL FORMAT

It is recommended that the trainee's first mock trial be conducted in-house with the attorneys as formal as possible. That is, the trainee should dress appropriately and maintain proper demeanor as would be expected in a trial setting. At a minimum, there should be individuals acting as prosecutor and defense to ask direct and cross examination questions. The trainee should provide answers and explanations as if speaking in the presence of a jury. Following the completion of the mock trial, those present may provide constructive criticism regarding the trainee's performance. The trainee may be asked to provide clarification statements either orally or written based on his/her answers during the mock trial. The final outcome of the mock trial will be satisfactory or unsatisfactory. If the panel present at the mock trial determines that the trainee's performance was not satisfactory, the trainee will be referred to the Laboratory Director.

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